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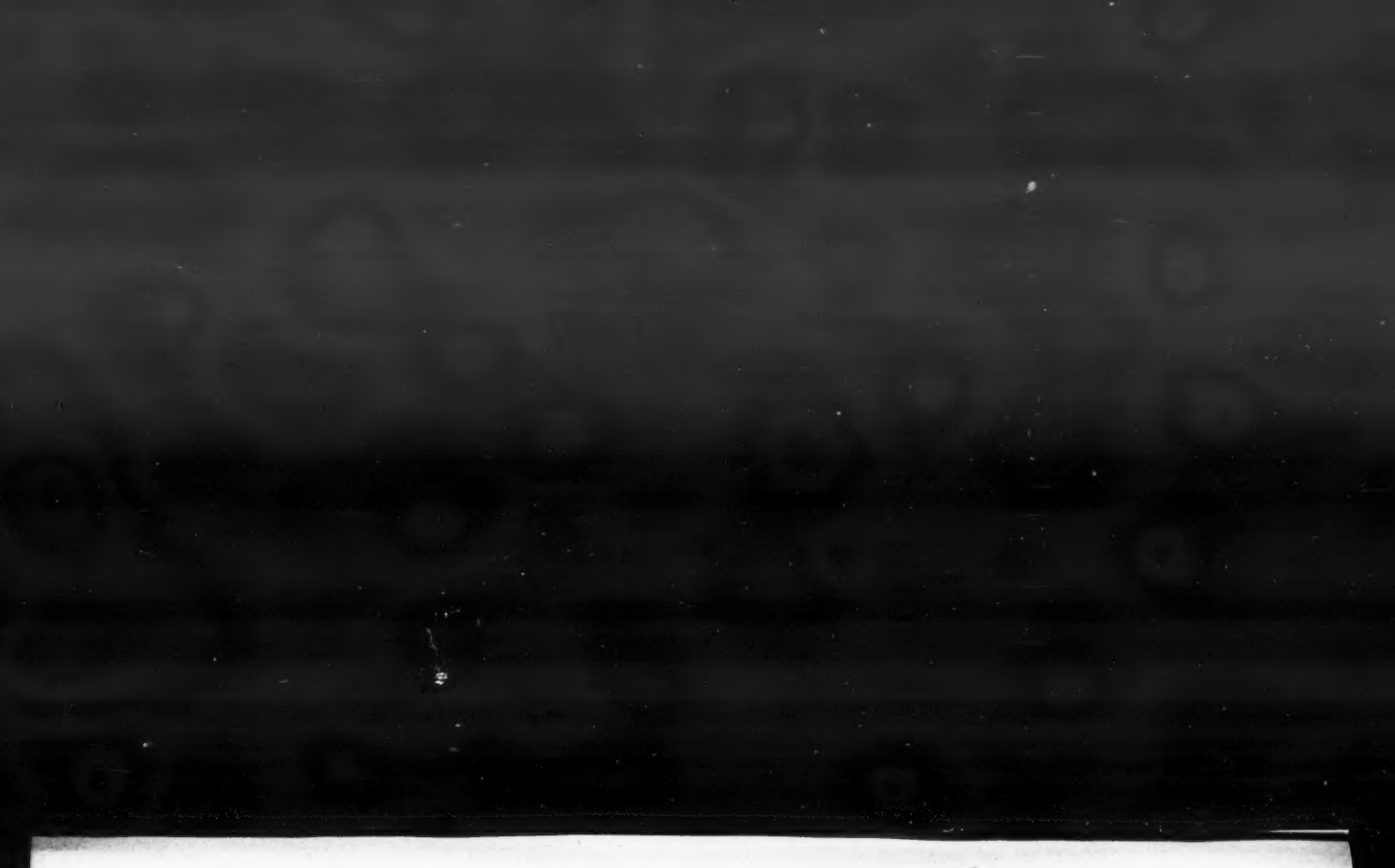


THE

AMERICAN JOURNAL OF SCIENCE

[FOURTH SERIES.]

ART. XVIII.—*On the Gas Thermometer at High Temperatures;* by LUDWIG HOLBORN and ARTHUR L. DAY.

[Communication from the Physikalisch-Technische Reichsanstalt, Charlottenburg, Germany.]

THE high temperature measurements which have been undertaken by various observers with the gas thermometer are not yet free from considerable disagreements.

Violle* determined the melting point of gold to be 1035° C., a value which he afterward increased to 1045°; Barus's† determination for the U. S. Coast and Geological Survey was 1092°, and one of the authors of this paper has published with W. Wien‡ the value 1072°.

In all three cases the melting point was not directly determined with the gas thermometer. Violle ascertained first the specific heat of platinum with the gas thermometer and then by calorimetric methods the melting point of gold, while in the last two instances a thermo-element was compared with the gas thermometer, after which it was used for the actual melting point determination.

Neither the indirect method, nor the inherent experimental difficulties which stand in the way of all investigation at high temperatures, sufficiently explain the differences mentioned. Especially recently, the relative measurement of high temperatures electrically has become less difficult and more accurate, as a consequence of which the variations of the temperature scales of different observers among themselves is the more unpleasantly conspicuous and disturbing.

* Violle, C. R., lxxxix, 702, 1889.

† Barus, Bull. U. S. Geolog. Survey, 54, 1889; Proc. Am. Acad., xxvii, 100, 1892.

‡ Holborn & Wien, Wied. Ann., xlvii, 107, 1892, and lvi, 360, 1895.

With the idea that these differences were, in part at least, due to the construction and handling of the gas thermometer itself, the first step toward the establishment of a new scale was thought to lie in the direction of an investigation of the principal instrument involved. The results of such an investigation are contained in this paper.

There is no doubt that the principal difficulty lies immediately at hand, viz., in providing a suitable bulb to hold the expanding gas. Since Deville and Troost* proved that hydrogen would pass through the walls of a platinum bulb at high temperatures, observers in general have turned to porcelain as the only practicable material, although the results obtained by Becquerel† with the Pouillet platinum bulbs compare by no means so unfavorably with the determinations above quoted, and the discrepancy between the Becquerel values and those obtained at about the same time by Deville and Troost was sufficiently explained by the dissociation of the gas (iodine vapor) chosen for the observations.

Among recent observers Violle, following the precedent of Deville and Troost, used porcelain bulbs glazed inside and out, while Barus, as well as Holborn and Wien, adopted those glazed only on the outside. The limits of usefulness of the bulbs with inside glazing must be reached when the glaze begins to melt, i. e. at about 1100°, otherwise vaporization is certainly to be feared. The porcelain walls themselves hold out longer, in fact the bulbs without inside glazing used by Holborn and Wien were used up to 1450°, but of course at such a temperature the pressure inside and out must be approximately the same, that the already soft walls may not be deformed thereby. At these temperatures porcelain bulbs only remain air-tight by virtue of the glazing—unglazed porcelain is not air-tight.

Brief review of investigation.—Our experiments up to a temperature of 550° were made with bulbs of Jena "borosilicate" glass No. 59^{III} and hydrogen as the expanding gas; then porcelain bulbs glazed inside and out were tried up to 1100°, first with hydrogen and afterwards with atmospheric nitrogen: next porcelain bulbs with outside glazing only were heated above 1200° with nitrogen; and finally a platin-iridium bulb, which when heated electrically proved entirely free from the disadvantages of porcelain, was used with chemically pure nitrogen over the whole range of temperature up to 1300°.

Toward the close of the observations we also had the advantage of entirely new manometric apparatus especially adapted to our purposes, in which the volume of unheated gas was much reduced and the use of low pressures in the bulb made possible.

* Deville and Troost, C. R., lvi, 977, 1863.

† E. Becquerel, Ann. de Chim. et de Phys., III, lxviii, 49, 1862.

The glass bulb proved to be exceedingly well adapted to the purpose up to 500°, showing no appreciable change in the zero point after being repeatedly heated, while with porcelain, even when nitrogen was afterward substituted for the hydrogen, the zero point did not remain constant, but rose slightly after each heating, denoting an *increase* in the gas pressure. Furthermore, the expansion-coefficient of the gas between 0° and 100° often showed changes larger than could be accounted for by the errors in observation.

Such changes, even when they remain small and the temperatures measured with the bulbs agree fairly among themselves, are persistent and disturbing factors in the measurement of high temperatures.

Another misfortune arises from the fact that porcelain is not a definite chemical compound, and bulbs from different sources may behave quite differently; even bulbs ordered at different times from the same source have the same unfortunate tendency.

These, with the above mentioned fact that bulbs glazed on the inside do not admit of use above 1100° at all, and the unglazed ones only with difficulty and under conditions of greatly diminished sensitiveness, led us to direct our attention once more to metallic bulbs and to investigate the behavior of a platin-iridium cylinder under the above conditions.

From the results thus far obtained we do not hesitate to say that in our opinion such a bulb is certainly to be preferred to porcelain for the higher temperatures.

This bulb filled with nitrogen and heated to 1300° showed a constancy of the zero point and coefficient of gas expansion such as we have only been able to obtain from glass bulbs at comparatively low temperatures. This result is, however, in no small degree dependent upon arrangements for heating by electricity and the consequent freedom from all sorts of combustion products, which if present, as we observed repeatedly, pass rapidly through the bulb wall, even against an excess pressure within.

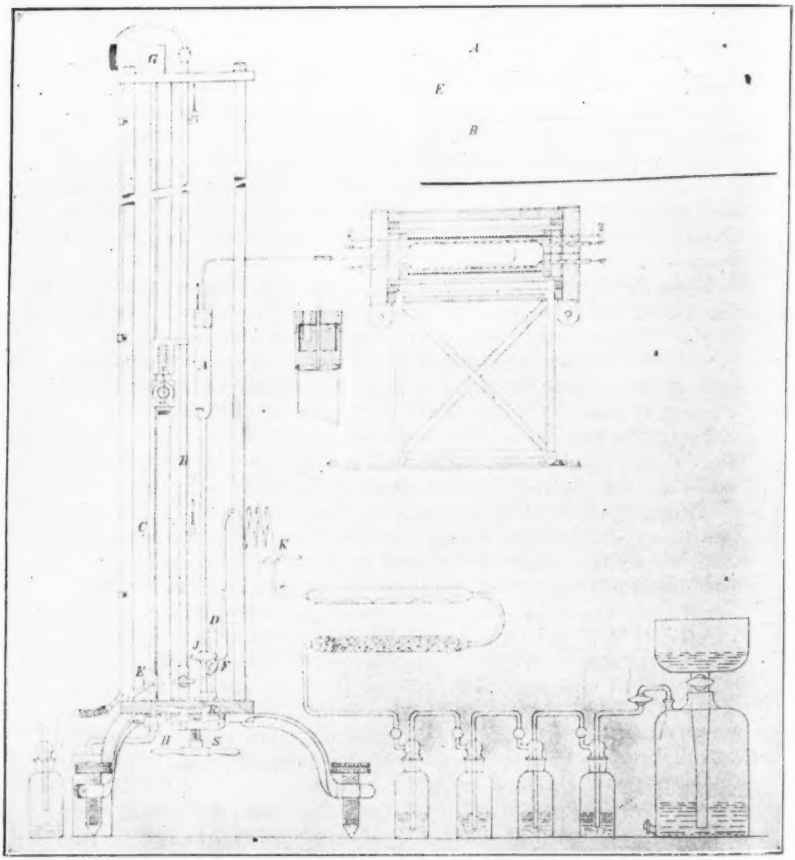
For comparison of the gas-thermometer readings among themselves, they were referred to a platinum, platin-rhodium thermo-element observed simultaneously.

For temperatures up to 750° bulb and thermo-element were dipped into a bath of nitre and in the vicinity of 900° a zinc vapor bath was tried. As no bath is yet available for temperatures above that point which does not seriously injure the walls of the bulb, the remaining temperatures were attained electrically by heating a coil of nickel wire wound upon a thin tube of porcelain or clay. The last proved the most convenient of all both for producing and maintaining constant any temperature within the range of the present series.

1. Gas Thermometer.

For the experiments with the gas thermometer the "constant volume" principle only was used, in order that a nearly uniform sensitiveness might be maintained throughout the

1



entire range of temperatures. As manometer we had at first at our disposal an apparatus constructed by Fuess (Berlin) and used and described by Wiebe and Böttcher,* a few necessary changes being made to adapt it to our purposes. Afterward a second similar instrument from the same workshop was used, a drawing of which (fig. 1) is here given.

* Wiebe and Böttcher, Zeitschr. für Instr., x, 18, 1890.

The manometer consists of two upright tubes, A and B, which dip into a closed iron reservoir, R, from which mercury may be drawn off through the steel cock, D, and let in from a storage reservoir, G, at the top, by way of the tube, C, and a second steel cock, E. Small changes in the height of the mercury column such as were necessary in the final adjustment for reading may be effected by means of the powerful screw, S, working upon a thin sheet-steel plate which forms the bottom of the reservoir.

The shorter tube is 80^{cm} long up to the metallic point where the adjustment to constant volume is made, for in order to avoid pressures much too great for the strength of the bulb at high temperatures, the pressure at 0° must be chosen small.

The adjustment to constant volume was made in the older instrument at the end of the platinum capillary which constituted the connection between the manometer and the bulb, and which was melted directly into the top of the shorter tube (corresponding to A). This arrangement, which we at first retained, left the "unheated space" unnecessarily large, which for our purpose was a decided disadvantage. The influence of this "unheated space" (the space between the surface of the mercury in the manometer and the bulb, filled with gas like the bulb but not heated with it) upon the resulting temperature increases much more rapidly than the temperature itself. We have consequently since then adopted the arrangement of Chappuis* and closed the short tube (A) by a carefully fitted metallic cap with polished under surface. This cap was fastened upon the tube with sealing wax and carried near the middle, where the capillary tube opened into the glass tube, a metallic point $0{\cdot}4^{mm}$ long to which the mercury column is brought at each reading. In this way the space above the meniscus was reduced from 2^{ccm} to $0{\cdot}4^{ccm}$ in a tube of 2^{cm} diameter. The metal point, intentionally rounded off in order to allow of more accurate setting, was made of nickel, as was also the entire cap, in order that it might not become amalgamated by contact with the mercury.

On the upper side this cap was hollow and was originally intended to contain mercury; at the last, however, we preferred to fit into the space a massive block of copper bored with a cylindrical hole to admit the bulb of a mercury thermometer. In this way the temperature of the space above the mercury column (A) is more accurately determined than if the thermometer were hung at the side.

We had, to be sure, in the old instrument a thermo-element within this space itself, the wires being passed in through the

*Chappuis, Trav. et Mém. du Bureau internat., vi, 28, 1888.

glass; the observations made with it also gave good results but the arrangement was inconvenient and awkward. At a distance of 161·61^{mm} below the nickel point, the tube contained a second point of colored glass projecting out from the wall of the tube itself, for purposes of volumetric measurement. The volume included between the points was 50·723^{ccm}.

The longer tube (B) was 2^{m} long and its inside diameter 12^{mm}. Its upper end is connected by means of a ground glass joint and a metallic return tube with a wash bottle containing phosphoric pentoxide, thereby assuring dry air above the mercury column.

To change the gas in the bulb a water aspirator could be attached to the wash bottle to secure the diminished pressure necessary for drawing the mercury from the shorter tube (A) when the pressure in the bulb was low. When the mercury is thus drawn below the three-way cock, J, this may be turned and connection made through a second similar cock, K, either with the mercury pump or the gasometer and drying tubes. All connections were made with glass or sealing wax and tested from time to time to see that they were and remained absolutely air-tight. Rubber connections were conscientiously avoided.

The temperature of the mercury in the manometer was determined by the thermometer fitted into the cap above described and three other similar ones, whose bulbs dipped into short tubes containing mercury placed directly beside the long manometer tube at proper intervals and giving the temperature at the corresponding height in the long tube with sufficient accuracy.

The oven being always in a separate room, the changes of temperature about the manometer were very small and the time between observations necessary for regulating the oven temperature to the required degree of constancy, always sufficient to enable the thermometers to indicate any small changes which may have existed.

When mercury was let into the apparatus from the reservoir (G) at the top, more or less air bubbles always came with it, which collected in the main reservoir (R), having no means of escape. To avoid this in the new instrument, a short tube, D, with a glass cock, F, was introduced into R at its highest point, while the manometer tubes extended much deeper, thereby allowing the air to be drawn off at any time without endangering the gas in the bulb—a matter of the highest importance.

The manometer scale was ruled in millimeters for a length of 1·87^{m}, and carried upon a slider the knife-edge and a micrometer screw and vernier reading intervals of 0·02^{mm} directly. The largest scale error amounted to 0 04^{mm}.

The platinum capillary tube serving to connect the bulb with the manometer was soldered at one end into the metallic cap before mentioned and at the other attached to the stem of the bulb with sealing wax, this arrangement proving quite as trustworthy and convenient as a soldered joint. The sealing wax could be kept cool with water if necessary. The length of the capillary tube was much reduced in the new instrument (from 1·37^{m} to 0·85^{m}), an improvement rendered possible by the electrical oven, which could of course be much better protected from radiation than a gas heater. Under the new arrangement a glass partition was amply sufficient to separate oven and manometer, a service for which before a brick wall had been deemed necessary. That the gas pressure in bulb and manometer may come quickly to a condition of equilibrium, the capillary tube should not be chosen less than two-thirds of a millimeter in diameter.

This flexible capillary also enabled us to move the thermometer bulb about at will for determining ice and boiling points after each heating; we found it, however, better to move it as little as possible, and in our later experiments the oven was mounted upon a framework provided with wheels and moving upon a track which enabled it to be pushed aside when the bulb was taken out, and ice and boiling point observations to be made at the same point by appropriately constructed apparatus. By this arrangement the amount of bending of the capillary required was very small.

Ice and boiling point observations were made after each heating during these experiments, not so much for their value as constants of the instrument and for the temperature determination as to determine any change which the measuring gas might have undergone at the higher temperatures.

The temperature as observed with the air thermometer was calculated according to the following formula:

$$H_{\circ}\left(1+\frac{v_{,}}{V_{\circ}}\cdot\frac{1}{1+at_{,}}\right)=\frac{H}{1+at}\left(1+3\beta t+\frac{v_{,}}{V_{\circ}}\cdot\frac{1+at}{1+at_{,}'}\right)$$

where

$H_{\circ}$ = Pressure at 0°.
H = " " t°.
$V_{\circ}$ = Volume of bulb at 0°.
$v_{,}$ = " " "unheated space" at the temperature of the room. (Changes in volume $v_{,}$ due to temperature neglected.)
$t_{,}$ and $t_{,}'$ = temperatures of "unheated space" with bulb in ice and at temperature t.
α = coefficient of expansion (technically *pressure coefficient* or *coefficient of expansion at constant volume*) of the gas between 0° and 100° (determined after each heating).
3β = cubical coefficient of expansion of the bulb.

The left side of the equation is a constant quantity for each filling if the zero point remains constant.

For the various bulbs used the values of V_0 and v, are quite different; the value in each case is therefore especially stated, and the percentage of effect which the "unheated space" has on the resulting t.

2. Electrical Measurement.

The measurement of the electromotive force of the thermo-elements was made by the "compensation" method. Cadmium cells served as standards of potential-difference.

The cold junctions of the thermo-elements were contained in glass tubes surrounded by melting ice.

A Siemens and Halske voltmeter was of great service in the regulation of the temperature of the oven.

3. Experiments with glass bulb.

A bulb of Jena glass No. 59^{III} was used for the measurements between 200° and 500°. The gas was hydrogen, chosen chiefly in order to see whether this gas, whose expansion is made the basis of modern thermometry, can be advantageously used in practice above 200°.

In these lower temperatures the thermo-elements do not form a good basis of comparison for the air thermometer; it is, however, perfectly possible to detect any change which the gas may have undergone during the heating through changes in the ice point or coefficient of expansion.

Dimensions of Bulb.—The glass bulb had the form of a cylinder with rounded ends, 16^{cm} long and $3{\cdot}5^{mm}$ outside diameter. The stem was $40{\cdot}0^{cm}$ long, $0{\cdot}75^{mm}$ in inside diameter, and bent to a right angle nearly in the middle.

The volume as measured at the beginning was $166{\cdot}53^{ccm}$, and at the end $166{\cdot}43^{ccm}$, the determination being made by filling with distilled water and weighing. The bulb had already been seasoned by repeated heatings to 500° before the first determination was made.

Unheated Space.—The temperature of the stem, as well as of the platinum manometer capillary, was determined by mercury thermometers placed at intervals close by and from whose readings an integral temperature of the unheated space, in this case amounting to $2{\cdot}701^{ccm}$, was obtained. This value is the result of a volumetric determination made at the beginning of the experiments. A determination by weighing with water at the close gave $2{\cdot}668^{ccm}$.

Heating arrangements.—For all the observations with the glass bulb a nitre bath was used, heated by Bunsen burners. The bath was contained in a cast iron kettle 34^{cm} high and 18^{cm} in diameter, provided with a cover through which the thermometer bulb and the porcelain tubes containing the thermo-element were passed, the whole being protected from radiation by a thick outer cylinder of fire-clay. The bath was stirred continually by a turbine wheel driven from outside. Inasmuch as the bath when cold solidified, the kettle was first heated to 250° empty and the previously melted nitre poured in through a funnel. At the close of the day's measurements the nitre was drawn off while still hot with a siphon.

Hydrogen.—The hydrogen used was generated electrolytically from dilute sulphuric acid, and before being admitted into the bulb was passed through two wash bottles containing alkaline solution of pyrogallic acid to remove traces of oxygen present, and drying tubes containing calcium chloride and phosphoric pentoxide. Failure to take this precaution always resulted in a fall of the ice point after heating, amounting to from 3 to 5^{mm} mercury pressure. This difference diminished with subsequent heatings (the heatings were each about five hours long), as may be seen in the following instance:

	Gas pressure at 0·0°.
Bulb freshly filled	567·80^{mm} Hg.
After 1st heating (to 510°)	564·63 "
" 2d " " 530	564·15 "
" 3d " " 500	563·61 "
" 4th " " 540	562·83 "
" 5th " " 540	562·56 "

The phenomenon is to be attributed to a gradual combining of the hydrogen with the oxygen present and occurs even when the temperature is not carried above 400°. Before the final filling the bulb was always evacuated at a temperature of 500° and allowed to stand for several hours, being "rinsed" from time to time with dry hydrogen. The generating apparatus and connections were perfectly air-tight and remained so throughout.

Expansion of Bulb.—For the glass bulb 3β is assumed equal to 0·000018,* a value determined for the temperature range 0°–100°. The "unheated space" affects the result (t) 2·6 per cent at 200° and 4·1 per cent at 500°.

*Thiesen, Scheel and Sell, Wissensch. Abhandl. d. Phys.-Tech. Reichsanstalt, ii, 129, 1895.

TABLE I.

Bulb (No. 1).—Jena glass No. 59^{III}. Gas—Hydrogen.

$V_0 = 166{\cdot}38^{ccm}$. $v_i = 2{\cdot}701$. $3\beta = 0{\cdot}000018$.

After filling.	H_0	a	t	e_1	Obs.-Calcul.
1st day	597·17	0·003657			
2d "	597·18	0·003658			
4th "			397·1°	3072	−0·3°
			393·1	3043	−1·2
			444·2	3485	−0·2
			518·4	4230	−0·8
5th "	597·17	0·003661			
6th "			342·4	2573	−0·7
			396·1	3073	−1·4
			458·1	3647	−0·5
			458·1	3656	−1·2
			487·3	3930	−0·8
7th "	597·19	0·003661			
Bulb freshly filled.					
1st day	638·87	0·003656			
2d "			309·3	2275	−0·7
			365·8	2787	−0·8
			406·8	3156	+0·8
			406·3	3158	−0·3
			462·1	3691	−1·0
			510·0	4147	−0·6
3d "	638·86	0·003656			
6th "			332·0	2470	+0·3
			371·9	2835	+0·1
			407·2	3163	0·0
			452·2	3586	+0·1
			502·5	4071	+0·3
7th "	638·72	0·003656			
8th "			311·4	2286	+0·2
			350·6	2640	+0·1
			405·5	3145	+0·3
			460·2	3653	+1·0
9th "	638·72	0·003655			
10th "			251·4	1763	+0·1
			250·7	1757	+0·1
			302·4	2201	+0·7
			413·8	3219	+0·7
			465·5	3708	+0·6
			510·7	4144	+0·4
11th "	638·67	0·003654			
15th "			234·8	1624	0·0
			254·2	1783	+0·6
			275·5	1967	+0·5
			551·3	4546	−0·2
			529·4	4322	+0·7
			514·7	4181	+0·5
16th "	638·26	0·003656			

Table I gives the results obtained from two separate hydrogen fillings between 200° and 500°. It contains the ice point (H_o), coefficient of expansion of the gas between 0° and 100° (a), the temperature as observed with the gas thermometer and calculated according to above formula (t), with the corresponding E. M. F. of the thermo-element T_1 in microvolts (e_1).

For this element a curve has been drawn based upon the mean of all the observations made with it between 200° and 1150°. Its form is given by the following table.

TABLE II.

Thermo-element T_1.

	Microvolts.		Microvolts.
200°	1334	700°	6044
250	1752	750	6566
300	2186	800	7096
350	2635	850	7634
400	3096	900	8180
450	3566	950	8734
500	4044	1000	9296
550	4531	1050	9863
600	5027	1100	10434
650	5531	1150	11009

The last column of Table I ("Obs.–Calcul.") contains the difference between any particular observed temperature and the corresponding point of this curve in degrees. The mean value of this error for this set of observations amounts to $\pm 0{\cdot}64°$ and is to be charged rather to the thermo-element than to the gas thermometer.

The differences between the separate values of the zero point (H_o) and of the coefficient of expansion (a) are not greater with the first filling than the maximum error of observation, which in reading barometer and manometer might in unfavorable cases combine to amount to $0{\cdot}05^{mm}$. The ice point in the second series of observations showed a tendency to fall, the amount of which, if we may except the last heating when the temperature was forced up to 550°, was $0{\cdot}14^{mm}$ (maximum).

4. Experiments with porcelain bulbs glazed inside and out.

Bulbs.—Temperatures above 500° were next measured with a bulb of this type. These bulbs, in a form adapted for this purpose, cannot, on account of the fine capillary tube, be glazed by simply dipping a finished bulb in the glazing solution and burning. Deville and Troost succeeded in glazing their bulbs on the inside, only by preparing bulb and capillary in separate pieces and melting them together in the oxy-hydrogen flame.

Inasmuch as the bulbs prepared in this way are often not air-tight, we have preferred to take the completely moulded though not burned bulb lacking only the end piece E (fig. 2), cover the inside with glazing solution, set the similarly smeared end piece on and burn the whole at once.

This gives of course on the inside an unglazed ridge some 2^{mm} wide and the same high where the cap is set on, but this has given no trouble.

To obtain a perfectly glazed outside surface (very essential) the bulbs must be hung up for burning by the stem, free from contact with each other or surrounding bodies. If it is desirable that the stem be curved for dipping in a bath, the finished stem may readily be bent in the oxy-hydrogen flame according to requirements.

The bulbs used by us were made in the "Königliche Porcellan-Manufactur" Berlin and have the form given in fig. 2.

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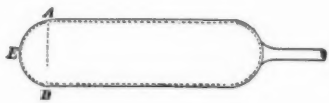


The length was about 12^{cm}, diameter $3{\cdot}7^{cm}$, thickness of the walls 2^{mm} and the volume approximately $100^{cu\ cm}$.

The length of the capillary stem was regulated according to our necessities, the original length being 60^{cm} and the diameter inside $0{\cdot}85^{mm}$. At the open end, it was bored out to a diameter of 2^{mm} to admit the end of the platinum capillary leading to the manometer; the joint was then made air-tight with sealing wax.

As coefficient of expansion the value obtained by Holborn and Wien for Berlin porcelain is adopted.

The "unheated space" with bulb No. II, mounted as here described, was $2{\cdot}837^{ccm}$. Effect on resulting t at 500°, 7·5 per cent; at 900°, 11·3 per cent.

Nitre Bath.—For a while the heating arrangements were retained as before, but above 500° the nitre bath decomposed more rapidly, in consequence of which it became strongly alkaline and its melting point sank; the outside glazing of the bulb was also considerably affected but remained air-tight throughout the observations.

The highest temperature attainable under these conditions is about 750°, but the mechanical arrangement for stirring could not be used above 600°. With an especially powerful heater and a smaller bath an attempt was made to carry obser-

vations still higher, but at about 810° a violent decomposition set in and in a few moments the entire bath had boiled away.

Hydrogen in porcelain bulbs.—At first one of these bulbs (No. II) was used filled with hydrogen, as in the case of the glass bulb; here, however, the bulb was evacuated at 700° instead of 500°, then rinsed as before with the dry gas. Table III contains two sets of results obtained under these conditions.

TABLE III.*

Bulb (No. II).—Berlin porcelain glazed inside and out. Gas—Hydrogen.

$V_0 = 101{\cdot}13^{ccm}$. $v_{,} = 2{\cdot}837^{ccm}$. $3\beta = 0{\cdot}0000132$.

After heating.	H_0	a	t	e_1	Obs.–Calcul.
1st day	529·02				
1st " (2)	529·03				
2d "			309·3°	*2258*	*+1·2°*
			410·1	*3179*	*+1·2*
			527·6	*4303*	*+0·8*
			611·4	*5119*	*+2·4*
4th "	527·56				
4th "	527·58	0·003669			
5th "			407·9	*3158*	*+1·3*
			521·9	*4234*	*+2·6*
			601·8	*5015*	*+3·0*
			708·6	*6100*	*+3·2*
6th "	525·36	0·003672			
7th "			499·1	*4018*	*+1·8*
			609·6	*5093*	*+3·0*
			653·3	*5527*	*+3·7*
			729·3	*6310*	*+3·7*
8th "	522·48	0·003678			
Bulb freshly filled.					
1st day	532·03	0·003665			
3d "			536·6	*4393*	*+0·7*
			613·6	*5142*	*+2·2*
			677·6	*5777*	*+3·5*
			725·2	*6269*	*+3·6*
4th "	529·37	0·003675			

After each heating a considerable fall in the ice point (H_0) is noticeable and an increase in the coefficient of expansion of the gas. This seems to point to a chemical action at the higher temperatures between the hydrogen and the walls of the bulb and the generation of water vapor.

Nitrogen.—For the reason named, the use of hydrogen at high temperatures was abandoned and nitrogen substituted. This was obtained from atmospheric air by passing it through from four to six wash bottles filled with an alkaline solution of pyrogallic acid, thence after continuing on through

* Values of *e* printed in italics were not used in the determination of Tables II and X.

the usual drying tubes it was used as before, first for rinsing at 700° and then for the definite filling. Especial precautions were here taken to free the bulb from hydrogen, the evacuated bulb standing the entire day at 700°, the pump being operated at intervals. The drying tubes were also evacuated before the nitrogen was admitted. Table IV gives the values obtained with the same bulb (No. II) and nitrogen, the heating being done as before in the nitre bath.

TABLE IV.

Bulb (No. II).—Berlin porcelain glazed inside and out. Gas—Nitrogen.

$V_0 = 101{\cdot}13^{ccm}$. $v_1 = 2{\cdot}837$. $3\beta = 0{\cdot}0000132$.

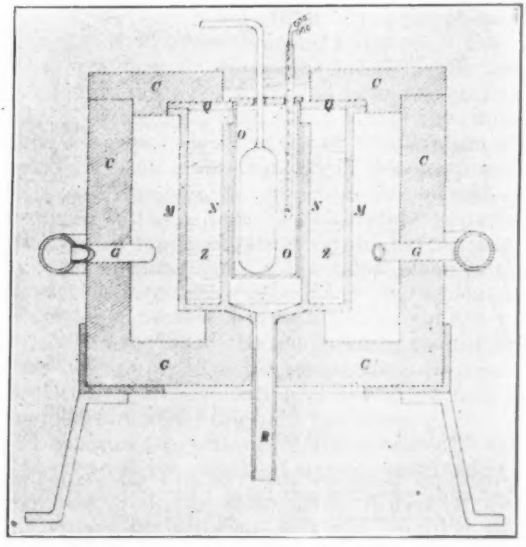
After filling.	H_0	a	t	e_1	Obs.–Calcul.
1st "	506·32	0·003677			
2d "			520·8°	*4249*	−*0·4*°
			606·3	*5092*	−*0·2*
			680·8	*5844*	+*0·2*
3d "	507·92	0·003681			
4th "			490·0	*3942*	+*0·6*
			573·7	*4760*	+*0.5*
			702·8	*6050*	+*2·2*
			706·9	*6088*	+*2·7*
6th "	508·54	0·003681			
Bulb freshly filled.					
1st day	432·87	0·003675			
3d "			280·4	2012	+0·3
			313·4	2301	+0·5
			512·3	4157	+0·6
4th "	432·62	0.003676			
6th "			506·0	4100	+0·2
			605·2	5087	−0·8
			633·8	5369	−0·2
7th "	432·41	0·003680			
8th "			612·2	5165	−1·6
			649·5	5537	−1·1
			690·9	5949	−0·1
			698·1	6015	+0·9
9th "	432·86	0·003680			
9th "			195·1	1295	−0·1
11th "			219·1	1488	+0·5
11th "			676·4	5789	+1·1
			673·9	5756	+1·8
14th "	433·09	0·003680			
15th "			576·7	4786	+0·9
			731·9	6366	+1·0
			735·1	6390	+1·9
16th "	433·61	0·003684			
Bulb freshly filled.					
1st day	417·29	0·003671			
2d "			813·1	7221	+1·4
			816·1	7253	+1·5

The first filling, in spite of the care exercised, still shows signs of the presence of hydrogen, afterward the changes in ice point and expansion coefficient become noticeably smaller, though the latter persists in a tendency to increase throughout.

The mean difference between the observed temperatures and those obtained from the curve (Table II) is $\pm$ 1·06°.

Zinc bath.—In order to continue these observations to a higher temperature an apparatus for boiling zinc was provided. The boiling kettle proper consisted of two concentric wrought iron cylinders, M and N (fig. 3), welded to a common bottom.

3



The ring-shaped space, Z, was filled with zinc while the inner cylinder, N, contained the bulb of the gas thermometer and the thermo-element, the latter protected as before by a porcelain tube. The whole was closed by a carefully fitted iron cover protected as much as possible by asbestos and fire-clay against loss of heat by conduction, and surrounded by a jacket of fire-clay pierced by a tube at the bottom where the zinc vapor passed off and the six blowpipes furnishing the heat. The latter were arranged in a spiral and directed tangentially against the zinc kettle.

It was unfortunately not possible to place the thermometer bulb and thermo-element directly in the zinc vapor on account

of the difficulty in packing them so as to prevent the vapor from escaping, consequently they were enclosed in a sheet iron cylinder made fast to the cover of the kettle. Around the outside of this cylinder the vapor circulated freely and took fire on coming in contact with the air at the end of the tube R.

Table V contains the results of two trials with bulb No. II in this kettle.

TABLE V.

Bulb (No. II).—Berlin porcelain glazed inside and out. Gas—Nitrogen.

$V_0 = 101{\cdot}13_6^{ccm}$. $v_{/} = 2{\cdot}83_7$. $3\beta = 0{\cdot}0000132$.

After filling.	H_0	a	t	e_1	Obs.–Calcul.
1st day	415·40	0·003669			
3d "			Heated to 700°.		
4th "	415·80				
4th "			804·2°	7219	−7·4°
			906 0	8293	−4·2
			920·3	8415	−1·0
			933·7	8564	−1·0
5th "	417·05	0·003669			
7th "			909·4	8304	−1·8
			914·8	8351	−0·7
			915·4	8354	−0·4
			916·6	8367	−0·3
			917·5	8373	0·0
			918·3	8382	0·0
			918·9	8387	+0·1
			919·4	8392	+0·2
			920·0	8393	+0·7
			920·4	8398	+0·7
8th "	417·29	0·003671			

An agreement between observed and calculated values, as will be readily seen from the table, is only to be obtained near the boiling point of the zinc, or after the generation of the vapor has begun to take place freely. The high temperature 933·7° on the first day was obtained by overheating the apparatus after the zinc was for the most part evaporated.

Electric Oven.—The second bulb of this sort (No. III) was heated electrically.

The coil (see fig. 1) consisted of bare nickel wire 2^{mm} in diameter wound upon a porcelain or fire-clay tube lying horizontally. The diameter of the tube was $4{\cdot}8^{cm}$ (inside), the length 35^{cm} and the thickness $0{\cdot}3^{cm}$ to $0{\cdot}5^{cm}$. The spaces between the separate turns of wire (of approximately the same width as the wire itself) were filled with clay which baked firmly to the tube, to prevent the wire from slipping and short circuiting when hot. In order to reduce the loss of heat into

the room to a minimum, the coil was first surrounded with one, later with two concentric tubes of clay, separated from each other as well as from the coil by a thin air jacket (1^{cm}). These spaces were closed at the ends and the tubes held in place by a tight packing of asbestos. Each tube was provided at the ends with a clay cover* fitting tightly, and between the covers was also left an air space of about a centimeter. Openings were filed in the covers to admit the stem of the thermometer bulb and the insulating tubes carrying the thermo-elements. This oven, while maintaining a temperature of 1000°, consumed about 1400 watts.

The thermometer bulb lay symmetrically in the oven tube resting upon three very small pieces of fire-clay, especial care being taken that it nowhere touched the oven. For each set of measurements two or three thermo-elements were used, stretched through the oven from end to end so that the junction could be brought to any point along the bulb whose temperature was required. The fall of temperature along the bulb amounted to not more than 6°, in the mean perhaps to half that. The results obtained from the several elements were all referred to the element T_n, a careful comparison with T_1 over the entire range of temperature having been made previously; the mean of these is the value given in the table. Inasmuch as this mean gave an excellent agreement with the gas thermometer in two so different sets of conditions (nitre bath and electric oven), and the temperature conditions within the oven can only improve as the temperature rises (compare § 6), no more accurate determination of the error due to variations of temperature along the bulb was made.

Table VI contains two sets of results obtained in this oven. In one respect the above described arrangement made a distinct gain over the previous one; with the bath the bulb had to be provided with a long bent stem for dipping into the kettle without unduly heating the sealed joint where the manometer capillary was attached; here a short straight one sufficed. The "unheated space" was thus materially diminished, and the error from this cause also. The effect of the unheated space upon t was here 9·7 per cent at 800° and 12·3 per cent at 1100°.

The temperature was not often carried over 1100°, only once a temperature of 1161·5° was reached, but directly following a large error in the ice point (1·30^{mm}) was recorded, which shows 1100° to be about the limit of the practical application of these bulbs. To be sure, differences in the zero point appear below this temperature; these in the calculation are distributed

* These clay covers (made by Haldenwanger, Charlottenburg), though perfectly able to withstand the high temperatures, were and remained of almost the consistency of chalk and could be filed or sawed at will.

among the observations of a particular series according to the *time*.

TABLE VI.

Bulb (No. III).—Berlin porcelain glazed inside and out. Gas—Nitrogen.

$V_0 = 106{\cdot}01_6^{ccm}$. $v_1 = 2{\cdot}80_7^{ccm}$. $3\beta = 0{\cdot}0000132$.

After filling.	H_0	α	t	e_1	Obs.–Calcul.
1st day	419·23	0·003672			
2d "			Heated to 1000°		
4th "	419·91	0·003669			
6th "			775·7°	6866	−2·7°
			826·0	7412	−3·4
			885·1	8034	−1·6
			932·1	8545	−0·9
			932·6	8553	−1·1
			934·5	8575	−1·2
			982·2	9128	−2·3
			984·6	9141	−1·7
			1058·8	9996	−2·9
			1058·3	9990	−2·8
			990·6	9203	−1·1
			897·3	8167	−1·5
			804·4	7162	−1·8
			700·6	6075	−2·4
7th day	421·12	0·003672			
9th "			Heated to 1050°		
11th "	421·61	0·003675			
13th "			783·1	6928	−1·1
			864·1	7797	−0·9
			924·5	8461	−0·9
			975·6	9044	−2·1
			1044·6	9824	−2·0
			1013·0	9462	−1·7
			1015·9	9496	−1·8
			945·0	8689	−1·0
			899·3	8184	−1·1
			833·0	7462	−1·1
14th "	422·33	0·003671			
16th "			820·2	7318	−0·5
			916·8	8352	+1·2
			1002·4	9323	0·0
			1097·9	10404	+0·5
			1127·8	10708	+0·5
			1061·8	9987	+0·9
			934·9	8559	+0·6
			861·5	7760	−0·1
18th "	422·89	0·003672			

Bulb freshly filled.	H_0	a	t	e_1	Obs.–Calcul.
1st day	412·66	0·003673			
2d "			803·2	7123	+0·7
			891·8	8094	−0·3
			1003·9	9311	+2·6
			1099·6	10400	+2·6
			1161·5	11128	+1·2
			1113·6	10574	+1·4
			1029·5	9616	+1·2
3d "	413·96	0·003671			
4th "			793·8	7043	−1·2
			901·6	8200	−0·2
			999·1	9292	−0·5
			1107·4	10517	+0·2
			1049·2	9844	+0·9
			938·1	8591	+1·0
			847·8	7610	0·0
5th "	413·99	$0·003675_5$			
6th "	413·90	0·003674			
8th "	413·97				

In order to be sure that the apparatus had remained air-tight, at the close of the observations several ice points were taken on different days. These agree within the limits of the error of observation. The mean difference between observed and calculated values of $t = \pm 1·52°$.

5. Experiments with bulbs glazed outside only.

The experiments with bulbs left unglazed on the inside did not differ materially from the preceding. The heating was all done in the electric oven. The old manometer was still used for the pressure measurements, but the unheated space was still further diminished by the use of a metallic cap for closing the short tube of the manometer at the top so that the correction at 500° amounted to only 4·7 per cent, at 1000° to 7·2 per cent, and at 1100° to 7·8 per cent of t.

The gas pressure was chosen considerably smaller than before, because the bulbs were to be heated above the melting point of the glazing; it was therefore necessary that the maximum pressure reached should not exceed the atmospheric pressure. The melted glazing, being thus continually pressed *into* the pores of the porcelain, held tight. Only a slight excess of pressure inside at this temperature suffices for the gas to filter through and escape.

Considerable difficulties were here experienced from the sticking of the melted glazing to the oven wall if the slightest contact occurred, in which case it was almost impossible to remove the bulb after cooling without cracking the glaze. It would then be no longer air-tight, nor in fact of any further

use for our purposes. In this way all the bulbs used were in a short time ruined.

TABLE VII.

Bulbs (Nos. IV and V).—Berlin porcelain glazed outside only. Gas—Nitrogen.

Bulb No. IV, $V_0 = 103{\cdot}47_8$ ccm. $v_{,} = 1{\cdot}77_0$ ccm. $3\beta = 0{\cdot}0000132$.
" V, $V_0 = 100{\cdot}21_0$ ccm. $v_{,} = 1{\cdot}66_4$ ccm. " "

After filling.	H_0	a	t	e_1	Obs.–Calcul.
			Bulb No. IV.		
1st day	171·02	0·003669			
4th "			545·0°	4480	0·0°
			641·5	5466	−2·1
			742·2	6505	−2·0
			843·9	7572	−0·4
			930·6	8523	−0·4
			1014·0	9485	−2·7
5th "	173·30	0·003663			
6th "			584·0	4886	−2·0
			693·8	5999	−1·8
			800·9	7126	−1·9
			896·3	8170	−2·8
			1010·2	9457	−4·0
7th "	173·26	0·003666			
			Bulb No. V.		
1st "	122·09	0·003667			
2d "			898·3	8145	+1·5
			906·3	8260	−1·0
			901·0	8238	−4·3
			Cont. to 1200°.		
3d "	125·01	0·003669			
4th "			948·8	8789	−6·1
			956·9	8884	−6·5
			957·4	8883	−5·9
			1069·7	10097	−0·8
			1080·9	10222	−0·6
			1077·1	10170	+0·2
			1080·2	10203	+0·4
			1149·0	10958	+3·4
			1150·4	10965	+4·2
			1149·2	10943	+4·9
			1157·2	11024	+5·9
			1036·3	9649	+5·2
			1029·2	9575	+4·6
5th "	126·21	0·003675			
6th "			900 8	8266	−7·0
			921·9	8505	−7·5
			987·1	9211	−5·4
			Cont. to 1225°.		
			997·0	9228	+3·0
			989·4	9160	+1·4

After filling.	H_0	α	t	e_1	Obs.–Calcul.
8th day	126·66	0·003678			
9th "			918·3	8435	−4·8
			923·9	8502	−5·3
			943·6	8704	−3·7
			1113·6	10630	−3·5
			1117·5	10664	−2·5
			Cont. to 1215°.		
			1066·4	10008	+3·7
			1066·8	10018	+3·2
			940·9	8622	+1·0
10th "	127·10	0·003679$_5$			
Bulb freshly filled.					
3d day	148·81				
5th "	148·85	0·003667			
8th "	148·88	0·003668			

In Table VII are given the results obtained with two of these bulbs. One (numbered IV in the table) served for measurements up to 1000° and was intended to furnish a control upon the results obtained previously from bulbs glazed on both sides.

The values given by the gas thermometer with this bulb are all too small when compared with the element T_1. It is nevertheless a matter of doubt whether this difference arises entirely from the nature of the bulb; it may be partly due to the change in the volume of the unheated space.

In regard to the changes in the zero point here noticed, it should be borne in mind that as the quantity of gas is here smaller, a small increase would produce a comparatively greater effect upon the pressure at 0°.

The other unglazed bulb (numbered V) was several times heated above 1200° and observed by rising as well as by falling temperature, as a result of which the differences between the values obtained and the normal curve are found to change sign during the heating, in fact to form a loop about the curve. The cause of this phenomenon we were not able to discover, for it was not possible, on account of the thin wall of the bulb, to heat it above 800° under vacuum, and further experiments with thick-walled bulbs could not conveniently be undertaken at that time.

A series of observations of the ice point and expansion coefficient was here also undertaken with a fresh gas filling at the close of Table VII, to see that the connections had remained tight.

In regard to the cause of the more or less systematic ice point variations throughout the observations, it is emphatically our opinion that it lies in the porcelain wall itself. The variations are always greatest after the first heating, so that quite

probably a chemical action or a setting free of absorbed gas is responsible for it. If porcelain at high temperatures were porous the change with each further heating should be as great as with the first. A change in the volume of the bulb cannot explain the phenomenon, for bulb No. III was weighed after the observations and its volume determined. The result showed an *increase* in volume amounting to 0·003, a result which would have an opposite effect to that under consideration.

Concerning the experiments of Teudt* tending to show a dissociation of atmospheric nitrogen at temperatures below 500° in which connection he has quoted experiments of Holborn and Wien, it may be said that we used the elsewhere mentioned platinum bulb at first with atmospheric nitrogen at temperatures considerably above 500° and detected no change in the gas whatever. We hope also later to investigate the behavior of porcelain bulbs with chemically pure nitrogen as well.

6. Experiments with the platin-iridium bulb.

The Bulb.—The platin-iridium bulb used was similar in form to the glass bulb first described and was made by the firm of Heräus in Hanau. It contained as much iridium as could be alloyed with the platinum and allow of its being properly worked—about 20 per cent. The length was 17^{cm}, outside diameter 4^{cm} and the thickness of the wall $0{\cdot}5^{mm}$. It was made from three pieces of sheet metal, the main cylinder and two rounded end pieces, welded together and giving of course a seam around each end and down the middle. These seams were afterward protected by a thick layer of platinum, melted and dropped on to the hot bulb—it then proved and has since remained perfectly tight. The stem where it was welded on to the bulb (welding seam also strengthened with platinum) was also made of the 20 per cent alloy for a distance of some 10^{cm} out from the bulb, where it was reduced to 5 per cent for the remaining 20^{cm} of its length. The 5 per cent alloy is much less brittle and, therefore, more convenient to handle. In outside diameter the stem was 2^{mm} and inside $0{\cdot}75^{mm}$. It was connected with the capillary tube leading to the manometer by means of a short overlapping glass tube sealed at both ends with sealing wax. It was possible by carefully heating to draw the sealing wax in between glass and platinum until the entire space around the ends of the two capillaries was filled, thereby adding as little as possible to the volume of the "unheated space."

* Teudt, Zeitschr. f. physikal. Chem., xxvi, 143, 1898.

The volume of the bulb at 0° was 208·222^{ccm} and the weight 308 grams. The final determination of the volume was made after the bulb had been several times heated to the highest temperature observed. The volume was chosen larger than in the case of the porcelain bulbs previously used in order to still further diminish the effect of the unheated space. *The required correction here was thus only 1·1 per cent of t at 500° and 1·9 per cent at 1100°.*

Before being attached to the thermometer the bulb was boiled for several days in concentrated nitric acid. When heated to 1100° or 1200° the outside surface turned black, a phenomenon probably to be attributed to the oxidation of the iridium. Afterward, when the temperature was carried up to 1300° the bright metallic color returned—indicating a reduction of the iridium oxide.

Gas filling.—Before filling the bulb with gas it was exhausted with the mercury pump and in this condition heated, at first to 850°, *afterward to 1300°*, the pump being operated from time to time throughout and a sharp vacuum maintained even at the latter temperature.

For the purpose of having an elementary gas for the final measurements, nitrogen was generated from ammonium nitrite and passed through four wash bottles containing the usual pyrogallic acid solution, then through drying tubes and into the hot bulb, which was then allowed to cool. When it had cooled down to the temperature of the room a further small quantity of gas was added or taken away until the pressure desired for the observations was obtained. This pressure was always so chosen that at the highest temperature to be measured, the maximum pressure exerted upon the walls of the bulb would not be above one atmosphere.

Electric Oven.—The oven was similar to that used in heating the porcelain bulbs; unfortunately, however, the tubes at our disposal upon which the coils could be wound were a few millimeters smaller than might have been desired, thereby giving rise to two sorts of difficulties. First, the irregularities of temperature within the oven made themselves strongly felt on the bulb of the thermometer, and secondly, inasmuch as the space between bulb and oven wall was very narrow the wires of the thermo-elements were with difficulty prevented from short circuiting through the metallic wall of the bulb. Their insulation was accomplished in these experiments by very thin porcelain tubes slipped on to the elements wherever such a contact was possible. At temperatures of 1000° and above, however, where the porcelain begins to act sensibly as a conductor, a leakage began to be noticeable from the oven coil into

the elements through the tube carrying the coil, the three bits of fire clay which served to hold the bulb in place in the oven, and the metallic bulb itself. The error due to this leakage within the range of temperature observed (to 1150°) was readily eliminated by reversing the current in the oven.*

For temperatures above this point a larger oven is being prepared. The oven as used up to the present is shown in cross section in fig. 1.

Temperature variations along the bulb. — The difficulty caused by the variations of temperature within the oven we succeeded in overcoming for the most part by varying the winding of the coil somewhat.

Instead of a uniform space between the separate turns of wire approximately equal to the diameter of the wire (2^{mm}) as we had previously used it, it was only so wound at the ends, the turns in the middle for some 10^{cm} being twice as far apart (4^{mm}) and coming gradually closer together toward the ends, i. e. roughly, a logarithmically wound coil.

Before the oven thus equipped was used for thermometric measurements the differences of temperature at various points within were determined as follows: Two thermo-elements were melted together at their junctions and drawn from point to point through the empty tube at various temperatures. Especially thin elements were chosen for this purpose, $0{\cdot}25^{mm}$ diameter instead of the usual $0{\cdot}6^{mm}$, in order to reduce the heat conducted away from the junction by the wires themselves to a minimum.

Table VIII contains the results of this trial for the old and the new oven coils, the dimensions of the tube being approximately the same for both. In the first case (uniform winding) the conditions improve with the higher temperatures, which seems to indicate that the tendency to a condition of equilibrium through radiation is stronger than the loss at the ends from conductivity. The presence of the platin-iridium bulb in the tube diminishes the irregularities some 3° at the lower temperatures, at 1000° scarcely any. The placing of fire-clay partitions in the tube close to the ends of the bulb also influences the results between 500° and 900°; between these limits the temperature as measured by the gas thermometer is raised some 3·5° without changing the temperature relation between the middle and end of the bulb; that is, the partitions affect only the radiation from the end surfaces.

* A simple reversal of the current is insufficient for correcting a large error from this cause, as the resistance of porcelain increases with the time of passage of the current, and one must, therefore, wait until the resistance is approximately constant. In the observations here presented the reversal of the current influenced the thermo-elements about 0·5° at 1000° and 1·5° at 1100°, and some five minutes were required to produce constant conditions.

TABLE VIII.

Variations of temperature within electric oven.

Distance of thermo-electric junction from middle-point of oven.	Fall in temperature as compared with center of oven.						
	Coil wound uniformly.			Coil wound logarithmically.			
	At 640°	At 840°	At 1040°	At 620°	At 820°	At 1010°	At 1150°
4·0cm to the right	— 3·4°	— 3·4°	−2·7°	+1·3°	+0·6°	−0·3°	−0·7°
7·5 " "	−14·7	−11·7	−8·7	+3·5	+2·1	−0·3	−1·8
4·0 " left	— 3·2	— 2·7	−1·9	+1·3	+1·2	+1·4	+2·0
7·5 " "	−14·4	−11·1	−9·9	+1·9	+1·8	+2·7	+1·7
10·0 " "				+0·1	+0·7	+2·4	+0·8

Table IX contains the results obtained with the platin-iridium bulb in the new form of coil, excepting the first two sets, which were made with the uniform winding and partitions in the tube close to the end of the bulb. For these two cases an integral value of the distribution of temperature along the bulb as measured at the time has been added as a correction. We lay no especial stress upon the agreement of these values with the following ones and give the results only because they happen to have been made with the same gas filling. Observations during which the oven current was reversed are indicated in the table by an asterisk (*).

The values for the electromotive force e_2 refer to the element T_2, which throughout the observations lay at the middle point of the bulb. The results obtained with the logarithmically wound spiral contain no correction for variations in temperature along the bulb, although, as may be seen from Table VIII, the ends of the bulb for the lower temperatures are somewhat overheated. The values in the last column (Table IX) give the differences between the observed temperatures obtained from the gas thermometer and those taken from a mean curve drawn through the plotted readings of the thermo-element T_2. *The mean value of the error thus obtained amounts to* $\pm$ *0·85.*

TABLE IX.

Bulb (No. VI).—Platinum with 20 per cent iridium. Gas—Nitrogen.

$V_0 = 208{\cdot}22_2{}^{ccm}$. $v_1 = 0{\cdot}87_0$. $3\beta = 0{\cdot}000025$.

After filling.	H_0	a	t	e_2.	Obs.-Cal.
1st day	286·44	0·003662			
4th "			625·9°	*5399*	*0·0°*
			626·7	*5404*	*+0·3*
			710·3	*6272*	*+0·6*
			815·9	*7408*	*+0·8*
			908·4	*8439*	*+1·1*
			1003·5	*9530*	*+1·7*
			1121·6	*10910*	*+4·5*
			1128·3	*11000*	*+3·8*

After filling.	H_0	a	t	e_{t2}.	Obs.–Cal.
5th day	286·35	0·003665			
8th "			705·3	*6252*	*−2·5*
			818·9	*7472*	*−2·0*
			915·7	*8565*	*−2·0*
			1015·6	*9710*	*−1·4*
			1130·0	*11074*	*−0·4*
10th "	286·39				
19th "	286·39	0·003662			
20th "			624·3	5389	−0·6
			809·4	7346	−0·1
			903·6	8387	+0·9
			1009·7	9614*	+0·8
			1107·2	10763*	+2·3
21st "	286·36				
22d "			511·1	4239	+1·1
			512·0	4255	+0·4
			550·6	4643	−0·4
			657·8	5743	−1·4
			702·0	6206	−1·5
			754·8	6765	−1·2
			856·2	7872	−0·8
			945·0	8882	−1·1
			996·6	9488*	−1·6
			1044·8	10036*	+0·2
			1047·2	10065*	+0·2
			1096·0	10645*	+0·8
24th "	286·30	0·003663			
24th "			540·4	4539	−0·1
			541·9	4556	−0·3
			614·3	5285	−0·4
			715·4	6332	−0·1
			811·6	7375	−0·5
			915·9	8541	−0·4
			1012·5	9653*	+0·3
			1100·0	10693*	+0·8
25th "	286·31				
25th† "			515·7	4291	+0·4
			615·1	5291	−0·2
			719·2	6371	0·0
			819·1	7452	0·0
			909·7	8449	+1·6
			1020·8	9748*	+0·5
			1127·5	11025*	+1·1
26th "	286·79	0·003662$_8$			
Bulb freshly filled.					
1st day	294·34	0·003666			
2d† "			528·3	4416	+0·2
			613·6	5277	−0·3
			1003·8	9547*	+0·6
4th "	294·33				

† At the close of the observations the oven was maintained for a considerable time at 1300°.

Table X gives the values of the E. M. F. for the element T_2.

TABLE X.

Thermo-element T_2.

	Microvolts.		Microvolts.
500°	4141	850°	7794
550	4633	900	8356
600	5135	950	8927
650	5647	1000	9507
700	6169	1050	10101
750	6700	1100	10703
800	7242	1150	11315

Expansion coefficient of platin-iridium.—In regard to the calculation of the temperatures given by the gas thermometer with the platin-iridium bulb, attention should be called to the fact that the round number 0·000025 has been adopted as the coefficient of volume-expansion of platin-iridium (3β).

This corresponds for a 20 per cent alloy to the figures given by Benoit* for observations upon pure platinum, pure iridium, and a 10 per cent alloy for temperatures 0°–100°.

It is probable that this coefficient is higher at higher temperatures, but few trustworthy observations are at hand. We have found only the papers of Le Chatelier† and Seliwanow.‡ The former gives as a preliminary result for a 10 per cent alloy between 0° and 1000° an expansion coefficient some 30 per cent higher than 0°–100°; the latter for platinum 0°–1650° gives a result only 11 per cent higher than at the lower temperatures. The temperature measurements is however, in the latter case, uncertain.

Table IX shows the values for the ice point (H_o) and coefficient of expansion (α) to be nearly constant. After the last heating, made with the first gas filling, a considerable increase in H_o will be noticed which, however, is solely due to the fact that the temperature was carried for the first time up to 1300°. Afterward when the bulb had been evacuated and freshly filled at 1300° a second heating to the same point showed no change in the ice point whatever.

In order to accomplish this result great care is necessary in the choice of the material used in the construction of the oven. We include herewith ice points from an earlier series of observations where a sudden and sharp increase occurred which could be foreseen during the progress of the heating. The outer mantle of the then new oven consisted of sun-dried

* Benoît, Trav. et Mém. du Bur. internat., vi, 190, 1888.
† Le Chatelier, C. R., cviii, 1096, 1889.
‡ Seliwanow, Journ. d. russ. phys. Ges., xxiii, II, 152, 1891.

(unburned) clay containing organic substances which burned when the bulb reached a temperature of 1000°, as could be readily detected by the odor in the room. The air thermometer at once began to go up and continued doing so for some minutes, though the thermo-element in the oven showed the temperature inside to be actually falling. This plainly showed that the gases of combustion were penetrating into the bulb in spite of an excess of pressure within of a full atmosphere. Further observations were suspended for the time and the oven allowed to cool, when the ice point was found to have risen 4·46^{mm}.

The zero points for the series follow:

Bulb freshly filled	296·67^{mm} Hg.
After heating to 1085°	296·66 "
After standing undisturbed for 18 days	296·66 "
After heating as above described	301·12 "

7. *Conclusion.*

We have then as a result of the foregoing investigation two curves based upon the thermo-elements T_1 and T_2 respectively. If we compare these we shall obtain the difference between the temperature scale measured with porcelain bulbs and that secured from the platin-iridium bulb. In Table XI this difference Δ is given in microvolts and in degrees.

TABLE XI.

	T_2-T_1	Δ	
	Microvolts.	Microvolts.	Degrees.
500°	93	4	0·4°
550	101	1	0·1
600	107	1	0·1
650	115	1	0·1
700	122	3	0·3
750	130	4	0·4
800	138	8	0·7
850	147	13	1·2
900	156	20	1·8
950	165	28	2·4
1000	174	39	3·3
1050	183	55	4·6
1100	192	77	6·4
1150	201	105	8·5

The difference, as will be seen, always has the same sign, that is, the scale founded upon the porcelain values always gives a somewhat higher value for the temperature than the other. In the lower part of the scale the Δ chances to be smaller than

the errors of observation ; even in the vicinity of 1000° it is not higher than the possible error in the value assumed for the coefficient of expansion of the bulb (3β). An increase of 10 per cent in 3β causes an increase of 1·5° at 1000° for the porcelain scale and 3·2° for the platinum. Above 1000° the values of Δ increase rapidly. The values obtained from the platinum bulb must be accorded the preference, however, not only on account of the constancy of ice point and coefficient of expansion but on account of the much reduced error due to the unheated space.

It is not impossible that at high temperatures a vapor is developed in the porcelain bulbs, in fact in the bulbs containing glazing this could quite naturally take place.

Our purpose is to continue the observations with the platin-iridium bulb to still higher temperatures and to investigate a second similar bulb which the firm of Heräus has kindly placed at our disposal. The coefficient of expansion (3β) and the effect of pressure at the higher temperatures will also be determined.

Dr. Hans Usener, who undertook this investigation with us and carried on for a considerable time the thermo-electrical observations, left it to accept a position at the German Government Torpedo Station at Kiel.

Charlottenburg, June, 1899.

ART. XIX.—*On the Flicker Photometer;* by OGDEN N. ROOD, Professor of Physics in Columbia University.

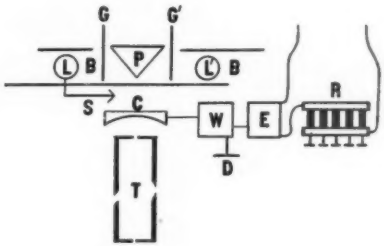
IN the September number of this Journal, 1893, I published an account of "A photometric method which is independent of color," the principle being, that when two colored surfaces are presented in rapid succession to the eye, the resulting flicker vanishes when they have the same luminosity. My experiments were afterward repeated and amplified by Dr. F. L. Tufts, who obtained confirmatory results.* In my paper I also remarked, that "there does not seem to be any reason why this process, with the aid of well known optical devices, should not be applied to ordinary photometric work, or to such as is carried on in the interior of the observing telescope of a spectroscope."

In 1896 Prof. F. P. Whitman devised a photometer with a revolving card-board disc, in which the proposed flicker method was utilized, and which gave satisfactory results.† On Nov. 17th, 1896, I read a paper before the National Academy of Sciences, in which were described four methods in which the flicker process could be employed photometrically: (*a*) by the use of convex or concave lenses oscillating at proper rates, (*b*) by a vibrating plane mirror, (*c*) by a revolving glass prism of small angle, (*d*) by a revolving plane mirror set excentrically on its axis. Apparatus embodying these ideas had at that time been constructed and used by me. In 1897 I constructed a complete photometer, using an oscillating convex cylindrical lens, and made many measurements with it. The instrument was briefly described in Science, June 3d, 1898, and a week later, in the same periodical, appeared a short account of some quantitative observations made with it on color-blindness. Since that time, the convex cylindrical lens has been replaced advantageously by one that is *concave*, and the paper prism previously used by one of plaster of Paris. The field has also been very much contracted, and other improvements introduced, particularly in the matter of uniformity of rate of oscillation and choice of such rate.

The general idea of the photometer is that the differently colored beams of light traversing its axis should illuminate the two surfaces of a rectangular prism, P, facing the eye, and that by the oscillations of a cylindrical concave lens, C, in front of the prism, its illuminated surfaces should alternately and in

* Transactions of the New York Academy of Sciences, xvi, 190–212, April, 1897.
† Physical Review, vol. iii, No. 16, Jan.–Feb., 1896.

rapid succession be presented to the eye. One incandescent lamp, L′, remains stationary; the other is moved by the observer till all flicker disappears, when a reading is registered on a fillet of paper, which is not inspected by the observer till the conclusion of the operation. In many of my experiments the light was colored by transmission through plates of colored glass, G, G′, and was almost spectral in hue. On a strip of board, B, 8 c. broad and 3·3 m. in length, graduated in centimeters, the incandescent lamps are placed, suitable guides keeping them in the axis of the instrument. One of the lamps can be moved by a long slender strip of wood, S, to which is attached a fillet of paper to receive the observations, which are impressed on it by a pin-point suitably arranged. The other lamp is stationed where in each case it is found desirable.



The prism, P, is fastened on the center of the long board at the common zero point of the two sets of graduations, and the most important thing about it is, that its two faces should meet in an invisible line; otherwise the juncture, not being illuminated, appears in the field as a vertical black line, and this will of itself furnish a faint flicker, that becomes apparent after the flicker from the faces of the prism has disappeared. Good prisms, free from this defect, can be made by casting plaster of Paris in a mould composed of glass plates, suitably ground on their edges. These plates can be removed from the plaster by the gentle heat of a spirit lamp, without damage to the dividing line between the faces of the still moist prism. When heat is applied, the plates fall off of themselves. With paper prisms, however well prepared, it is impossible entirely to get rid of the black dividing line, and the observer is obliged to rely on obtaining a minimum flicker.

With the above object still in view, it is best to use a *concave* cylindrical oscillating lens, which diminishes still further the bad effects of any want of perfection in the prism; that employed by me had a focus of 13 centimeters; its outline was square, the sides being 4 centimeters in length. It was placed

parallel to the line where the prism faces meet and also parallel to the axis of the instrument. Its distance from the prism was 16 centimeters.

The observing tube, T, had a length of 21 and a diameter of 4 centimeters. It was placed at right angles, both to the vertical axis of the prism and to the axis of the photometer. Both ends of it were furnished with diaphragms pierced by apertures 5^{mm} in diameter; the end next to the cylindrical lens should be brought as near to the lens as practicable. The visible field thus obtained is rather small, but quite sufficient, larger fields having been found to give inferior results, particularly in the hands of untrained observers.

In the apparatus as at present arranged a small electromotor, E, is used to give the cylindrical lens the proper oscillatory movement through the intervention of a train of toothed wheels, W. The speed of the motor is regulated in the following way: a current is employed more than sufficient to drive the motor at proper speed, and the strength of this current is reduced by the introduction of a variable resistance, R, composed of five glass tubes, each of which is 13 centimeters in length with an internal diameter of 2 centimeters. The tubes are filled with a mixture of coarse graphite and asbestos, which can be more or less compressed by the aid of five screws provided with plungers. As each tube carries only the fifth of the current, the rise in temperature is inconsiderable. The idea of employing the mixture for a variable resistance was borrowed from Dr. Hallock. In this way the speed of the oscillating lens can be regulated and kept constant for a long time. For the sake of convenience, a rotating card-board disc, D, is attached to W, and provided with a proper number of black and white sectors. Its appearance enables the observer to judge whether the proper rate of speed is being employed.

The photometer is most sensitive, that is the flicker is strongest, when the rate of oscillation is such that the two colored surfaces in the field are just fairly blended by the movement of the lens, and cannot be distinguished separately. The rate of lens oscillation usually employed has been about sixteen per second, more or less. When the illumination is feeble, low rates are preferable; when it is strong, higher rates are better, and some persons seem to have a general preference for rather high rates, which while diminishing the sensitiveness somewhat, also abolish flicker due to imperfection in the prism.

Finally, movable screens of black pasteboard are arranged around the prism, observing tube, and partially around the head of the observer, in such a way that all extraneous light is cut off.

The mode of making the observations is much the same as with ordinary photometers; by a preliminary experiment the position of flicker disappearance is approximately ascertained, and then the moving lamp is displaced till a distinct flicker is noticed; it is then brought up till this disappears, and the operation repeated in the reversed sense, both observations being recorded. From time to time wider excursions are made, to avoid the danger of "falling into a rut." Two fiducial marks are finally impressed on the fillet of paper, which is removed and read with a lens and millimeter scale. Persons trained in laboratory work usually obtain good results in their first trials; this was the case with Miss Furness, Dr. Dennett, Professor Hallock and some of the laboratory assistants. In other cases a certain amount of training was found necessary, which seemed to be due to the fact that the attention of the observer was allowed to wander from flicker-perception to color-perception, and to the changes in it due to the position of the movable lamp. With the small field now employed, there is no eye-fatigue, except that incident on looking steadily into any kind of tube for some minutes.

In order to furnish an idea of the degree of accuracy attainable, I give below sets of results obtained by myself when using three pairs of colors, red and blue, red and green, green and blue. The colored light was furnished by colored glass, and was to all appearance spectral in its purity. One face of the prism being illuminated with red light from the stationary lamp, the other received violet-blue light from the movable lamp, which was adjusted till the flicker vanished, and its mean distance from the edge of the prism obtained, ten observations being made. For certain purposes this operation was repeated on seven different days. Taking the mean distance of all seven results as correct, and as corresponding when squared to 100 per cent, I calculated the deviations from this in each of the seven cases in percentages.

Red and Blue.	Diff.	Red and Green.	Diff.	Blue and Green.	Diff.
Dec. 13	−0·26	Feb. 2	−0·89	Jan. 5	+3·5
" 16	+1·74	" 3	+2·60	" 16	+0·01
Jan. 5	+0·4	" 11	+1·10	" 17	−1·91
" 13	−0·26	" 15	−1·69	" 18	+0·84
" 16	−1·57	" 17	−0·35	" 24	−1·26
" 17	−0·26	" 20	−1·95	" 25	−1·26
" 18	+0·3	March 1	+1·29		

Similar results are also given above for the pair red-green and green-blue. No account is taken of possible variations in the

relative intensity of the two lamps, since such variation would at least not have tended to increase the accuracy of the results.

In order to exhibit the working of the instrument in the hands of other persons, I give below results obtained by six good observers, with each of whom I compared my own color-vision; they contain an element which furnishes a rather severe test of the correctness of the whole procedure that is entirely wanting in the tables above given. Each of the six persons was successively, for the time being, assumed to have standard color vision, viz: their perception of red, green and blue was taken as 100 in each case, and I determined my variations from it, both of us using the pairs, red-blue, red-green, and blue-green. In this mode of procedure it was possible to obtain two values for my perception of green as compared with theirs, a direct one founded on our joint experiment on the pair red-green, and an indirect one derived from our joint observations on the pair green-blue, my value for blue having first been obtained by a third joint experiment on the pair red-blue. If all the readings obtained by the person used as a standard and by the one undergoing examination are correct, then the direct and the indirect value for the green should coincide. Attention is again called to the fact that the indirect value is obtained by the use of an *intermediate* color. Below is a table showing the variations in percentages between these direct and indirect values of the green.

Dr. Dennett	−0·32	Mr. Parker	+1·77
Miss Furness	−1·04	Dr. Tufts	+3·17
Dr. Hallock	+0·41	Mr. Wade	−0·69

The general conclusion to be drawn from the numerical results given in this paper would seem to be, that the accuracy attainable with the flicker photometer, as at present constructed and using light of different colors almost spectral in hue, is about the same as with ordinary photometers using plain white light, or light of exactly the same color.

New York, May 8th, 1899.

Art. XX.—*A Quantitative Investigation of the Coherer;* by A. Trowbridge.

Since the very remarkable success of G. Marconi in the field of wireless telegraphy, a renewed interest has been felt by physicists in the coherer, the apparatus devised by Branley which has made wireless telegraphy possible.

Besides the original paper* of Branley there have been a number of communications on the subject of coherer action; in particular those of Dorn† and Aschkinass,‡ which have thrown much light on the subject. As yet, however, the experimental data are not complete enough for a perfectly satisfactory theory of coherer action to be formed, those already existing being seemingly inadequate to explain all the observed phenomena.

Even if we do not know just what takes place in the coherer itself, we know that the cause of the fall in resistance is primarily an electro-magnetic disturbance in the space surrounding the coherer. This disturbance will induce a static wave in a conductor whose direction in space coincides with the direction of the lines of electric force from the source of the electromagnetic disturbance. If this static wave is the cause of the lowering of the resistance of the coherer, then a static discharge from a Leyden jar, or electrophorus, through the coherer, should produce a like effect. This conclusion was verified by Professor Henry S. Carhart and myself while we were engaged a few months ago in testing the sensibility of coherers for telegraphic purposes. If this conclusion is warranted, then on the nature of the discharge and the quantity of electricity which goes through the coherer should depend the fall of resistance of the latter.

The present paper has to deal with the lowering of the resistance as a function of the quantity of electricity discharged through and the difference of potential on opposite sides of the coherer.

In order to test the supposed relation between quantity of electricity discharged and the fall of resistance in the coherer, I adopted the following arrangement.

By means of a charge and discharge key, a subdivided condenser could be charged from a large storage battery, the electromotive force of which could be varied at will from 2 to 70 volts, and then discharged through the coherer. The capacity at my disposal could be varied from 3·2 microfarad to 0·05

* Compt. Rendus, cxi.
† Wied. Ann., lxvi, p. 146.
‡ Wied. Ann., lxvi, p. 284.

microfarad, and this range, taken together with that of the storage battery, made it possible to vary the product $CV = Q$, within a comparatively large range, the upper limit of which, however, was only 224 micro-coulombs.

For the purpose of measuring the fall of resistance, a branch circuit containing a single storage cell (E. M. F.=2 volts) and a milli-ammeter was connected to the ends of the coherer. The limit of accuracy in reading the milli-ammeter was 0·001 ampere.

With a coherer of the type described by Marconi, I found, as I expected, an increase in conductivity with an increase in quantity of electricity sent through the coherer; however on increasing Q beyond a certain point, the corresponding increase in conductivity was very small; for the coherer in question, for example, the increase in conductivity was very rapid on increasing Q up to about 15 micro-coulombs, but beyond this point, doubling the quantity only produced a fractional change in the conductivity. It seemed as if the 15 micro-coulombs were enough to produce all, or nearly all, the lowering possible for the coherer in question and for the given charging potential (a point I will explain presently).

As the results obtained with the above mentioned coherer were not concordant enough for quantitative work, I adopted another type which I have since seen described in a paper by Branley,* in which he claims that it is more sensitive than the older form.

The coherer I used consisted of 21 bicycle pedal balls (diameter 3^{mm}) mounted in a horizontal glass tube of about the same bore, the end balls being soldered to lead-wires and the tube being provided with a device for adjusting the pressure of contact between the balls. The normal resistance of this coherer was about 2000 ohms, care being taken always to return to about this resistance by tapping the tube and adjusting the pressure before an observation was taken on the fall of resistance due to the discharge of the condenser. M. Branley used hard steel balls 12^{mm} in diameter—those first used by me were $9{\cdot}5^{mm}$ in diameter, but I found the smaller ones finally used to be much better suited to my purpose, at least, and I should judge that this would also hold true for telegraphic purposes.

However, I made no attempt at long-distance telegraphy with my coherer, having only satisfied myself that, at short range, the ball coherer behaved in all respects like a filing coherer.

The method of observation was the following: With a given charging potential the capacity was varied through the limits

* Comptes Rendus, No. 18, May, 1899.

above mentioned: for each capacity the mean of about 20 observations was taken on the lower limit of resistance attained by the coherer on discharging the capacity through it; this resistance being indirectly obtained by Ohm's law from the readings of the milli-ammeter.

As I have mentioned above, the upper limit, or normal resistance of the coherer was arranged to have a uniform value of about 2000 ohms.

Next, the charging potential was given another value and a like set of observations taken, and so on through the range of the charging potential.

It was at once evident that the lower limits of the resistance was not the simple function of the quantity of electricity sent through the coherer that I had expected. If this had been the case, I should have found the lower limit of the resistance the same for the cases $Q = C_1V_1 = C_2V_2 = C_3V_3$, etc. = constant. From my observations this was distinctly *not* the case.

I found that for Q = constant, the larger the charging potential—and hence the smaller the capacity—the lower was the final resistance or the greater the conductivity of the coherer after the discharge.

I have given the results of my observations on the ball coherer in curves 1 and 2. A table of the numerical values I obtained I have not given, since it would be of little theoretical import, as it would hold only for the coherer I used. The form of the curves given should, I believe, be similar for all coherers.

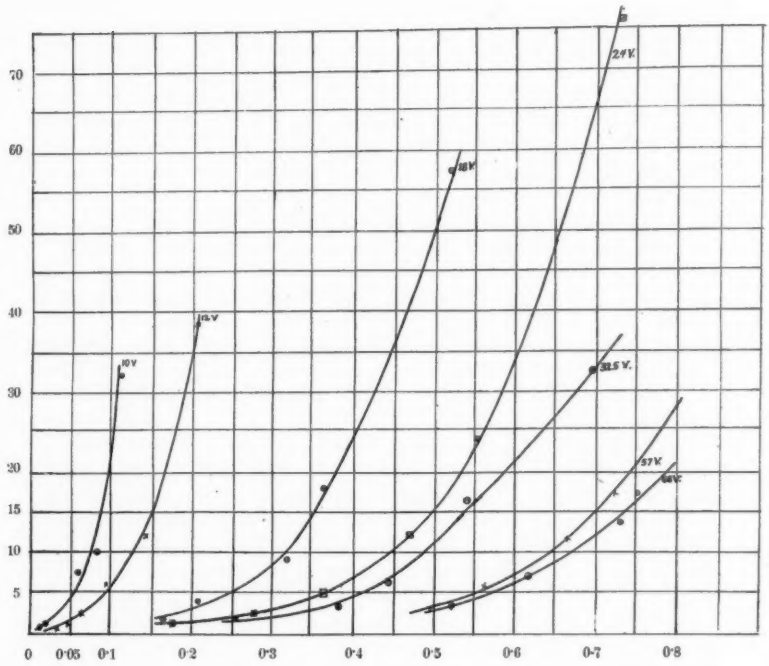
In curve 1, the abscissas represent the reciprocals of the lower limit of resistance of the coherer, or the conductivity; the ordinates, the quantity of electricity sent through. The seven different curves represent the change in conductivity as a function of the quantity for the seven different charging potentials used; the indices V=10, 12, 18, etc., denoting these potentials.

As will be seen, all the curves tend in a general way, as they should, towards a point on the conductivity axis, very near the origin; a point which would denote the reciprocal of 2000 ohms, the normal resistance. Also it is clear that the greater the charging potential, the more rapid the rise of the conductivity per unit increase in quantity discharged.

I was unable, with the coherer used, to get coherer action when the charging potential of ·8 volts and under was used. This point is brought out by the set of curves No. 2, in which the abscissas are the same as in curves No. 1, while the ordinates represent charging potentials. All the curves Q = constant cut the ordinate axis at a point V = 8·75 volts. This is, so to speak, the critical voltage for the coherer under

examination. Unless a potential difference between the terminals of the coherer of at least this value is produced by the electromagnetic disturbance, no coherer action will take place.

Probably every coherer has a critical value of the potential difference peculiar to itself, a coherer with a low value being preferable for telegraphic purposes. Owing to lack of time I have not been able to experiment with more than one coherer.



When we inquire into the nature of the discharge through the coherer which produces a lowering of resistance, the question arises as to whether or not it is of an oscillatory character.

From the theory of the oscillatory discharge of a condenser, we have a term

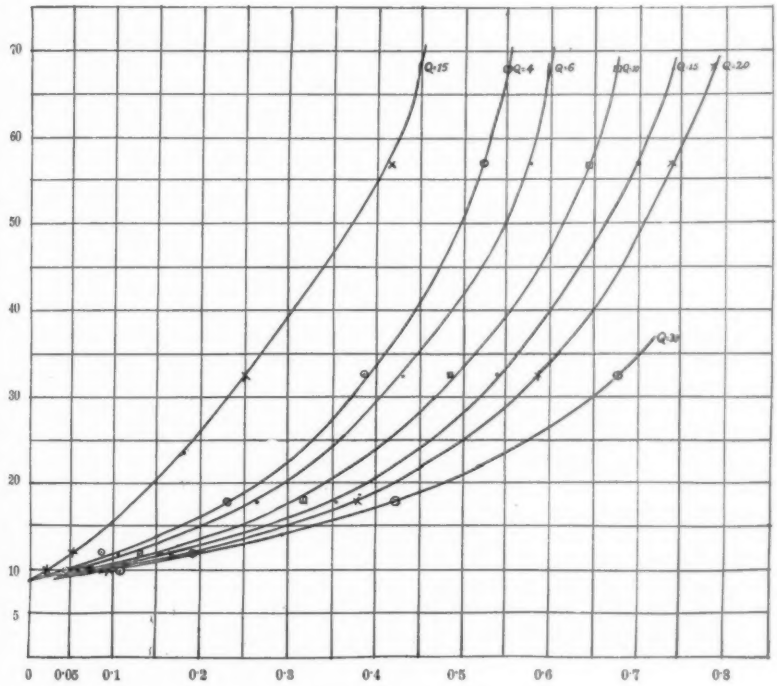
$$\sqrt{r^2 - 4\frac{L}{C}}$$

entering into the expression for the current strength I, where r is the resistance of the discharge circuit and L and C respec-

tively the self-induction and capacity. If $r^2 < 4\frac{L}{C}$, an imaginary quantity enters into the expression for I, this is the condition for a periodic discharge. If, on the other hand, $r^2 > 4\frac{L}{C}$, we have an aperiodic discharge.

Under the conditions present in my investigation, the latter case was certainly realized, for r normal was 2000 ohms, C

2



from 3·2–0·05 microfarad and L certainly very small, say less than 0·01 quadrant.

$r^2 = 4 \cdot 10^6$ ohm $= 4 \cdot 10^{24}$ cm. sec.$^{-1}$
C $= 0 \cdot 05$ microfarad $= 5 \cdot 10^{-17}$ cm.$^{-1}$ sec.^{2}
L = say 0·01 quadrant $= 10 \cdot 10^6$
$4\frac{L}{C} = 4\frac{10 \cdot 10^6}{5 \cdot 10^{-17}} = 8 \cdot 10^{23} < [4 \cdot 10^{24} = r^2]$

As L is certainly less than the value here assumed, the discharge circuit being a short straight copper wire, it is evident

that $r^2 > 4\frac{L}{C}$, or that the discharge which affects the lowering of resistance in this investigation was aperiodic. I tested this point experimentally in the following manner: the condenser was removed and the lead-wires from the storage battery to the ends of the coherer were provided with a key and a thin $\frac{1}{10}$ ampere fuse-wire in series. When the key was closed a steady current flowed through the coherer—the voltage of the storage battery was varied through a range of from 8–70 volts, and the lowering of the resistance of the coherer was again found to vary as the voltage. Owing to the element of time entering into the problem (the time elapsing from the closing of the key to the blowing out of the fuse), it was not possible to make quantitative observations on the fall of resistance. It was evident, however, that a steady current did produce this drop in resistance, that the drop was greater the greater the electromotive force of the storage battery, and further, as I had before noted, that for an electromotive force less than from 8–10 volts no coherer action took place.

Now we can regard the coherer with its vertical wire and earth connection, as it is used by Marconi, as part of the discharged circuit of a condenser whose plates are the vertical wire and the earth. A certain potential difference between vertical wire and earth is caused by the electromagnetic waves sent out from the Herz oscillator; this potential and the capacity of the vertical wire—earth condenser determine the quantity of electricity which is discharged through the coherer in the form of an aperiodic impulse and which produces the lowering in resistance necessary to wireless telegraphy. The potential difference on opposite sides of the coherer must be an inverse function of the distance between sending and receiving stations; if this distance is small, the potential difference being above what I have called the critical potential for the coherer used, if we increase the capacity of the vertical wire we shall increase the fall in resistance of the coherer, but if the distance is so great that the potential is at about its critical value, then an increase in capacity would lower the potential and so diminish or destroy the sensibility of the coherer.

This would explain Marconi's statement that at short distances a capacity consisting of a cubical tin box mounted at the top of the vertical wire, increased the sensibility of the coherer, but that he found it advisable to omit this capacity when telegraphing long distances.

It is also evident that one precaution must be taken when using a very sensitive coherer for wireless telegraphy. Since

a steady current will produce coherer action if the potential difference between the ends of the coherer exceed what I have called the critical voltage, and since the lower this critical value is, the more sensitive will be the coherer for long-distance work, care must be taken that the electromotive force of the battery which operates the relay in Marconi's arrangement, shall have a value less than the critical value for the coherer used.

If an electromotive force of about the critical value is used, we might expect to notice a confusion of the signals from the sending station due to the local circuit through the coherer producing an independent and quite irregular lowering of the resistance.

Everyone who has attempted wireless telegraphy has probably been annoyed by extra "clicks" of the instrument. I believe it is in many cases attributable to the cause just mentioned; in which case it could be done away with by so choosing critical potential of coherer, electromotive force of battery in coherer circuit, and sensibility of the relay, that the first should be higher than the second, while the third should be great enough to permit of a small absolute value of the other two.

In conclusion, I should like to take this opportunity to express my thanks to my colleague, Dr. Karl Guthe, for his kindly interest in the present investigation, and for numerous helpful suggestions.

Physical Laboratory of the University of Michigan, June, 1899.

ART. XXI.—*The Double Ammonium Phosphates of Beryllium, Zinc, and Cadmium in Analysis;* by MARTHA AUSTIN.

[Contribution from the Kent Chemical Laboratory of Yale University—LXXXVI.]

IT has been shown* that the composition of the phosphate of manganese thrown down by microcosmic salt from the solution of a pure manganous salt contains more manganese than belongs to the ideal ammonium manganese phosphate NH_4MnPO_4; and, further, that by acting with ammonium chloride in proper proportion the phosphate of manganese thrown down by microcosmic salt may be completely converted to the ideal ammonium manganese phosphate. Ammonium chloride, likewise, in the case of magnesium phosphate† tends to cause the replacement of the metal by ammonia. Indeed, the replacement here is readily carried so far beyond the point corresponding to the normal ammonium magnesium phosphate, NH_4MgPO_4, that the tendency to form a salt richer in ammonia and poorer in magnesium—perhaps something like $Mg(NH_4)_4(PO_4)_2$—must be recognized.

These facts suggested an investigation into the constitution of certain other ammonium phosphates with reference to their utility in analytical processes. Of the elements of Mendeléeff's second group, beryllium, magnesium, zinc, cadmium and mercury are capable of yielding double ammonium phosphates, while no such compounds of calcium, strontium and barium have been described. The solubility in ammonia of the double ammonium phosphates of the elements of the former category appears to increase as the elements of which they are compounds are removed in the series from the beryllium, and, while the same is true of the simple phosphates of members of the latter category, the extent of such solvent action is slight comparatively. According to the work recorded in the literature, calcium, barium, and strontium form individually a neutral tribasic phosphate or acid phosphates of greater or less degree of acidity according to the conditions of precipitation. In my experience where salts of these elements were precipitated either with ammonium phosphate or microcosmic salt in presence of varying amounts of ammonium chloride, or ammonia, or both, only the recognized phosphates were obtained. The effect of ammonium salts in presence of ammonia seemed to promote the formation of the tribasic salt in the case of calcium and strontium; barium tends to form the barium acid phosphate almost exclusively even in the presence of ammonium

* This Journal, vol. vi, 233. † This Journal, vol. vii, 187.

salts and free ammonia. No double ammonium phosphate of either calcium, strontium, or barium was produced under any condition. As is well known, mercury does form an ammonium mercury phosphate, but the salt is soluble to so great a degree in ammonia, ammonium chloride, and even in the precipitant itself, that nothing of any value for analytical work seemed likely to come from its study.

The Ammonium Beryllium Phosphate.

The ammonium beryllium phosphate has been described by Roessler* as a crystalline salt produced by boiling some time in ammoniacal solution the phosphate precipitated by ammonium phosphate, though the best results of this treatment failed to yield the ideal constitution of this salt, NH_4BePO_4. This same precipitate can not be obtained, Roessler further states, by using a sodium salt as the precipitant. In order to follow out this work of Roessler, a solution of beryllium chloride for use was prepared as follows: The pure beryllium chloride of commerce was dissolved in as little water as possible and treated for the precipitation of aluminum by etherial hydrochloric acid.† After filtering and evaporating from the filtrate the ether and a part of the hydrochloric acid, the beryllium was precipitated with ammonia, filtered to remove any members of the magnesium group, and washed free from ammonium chloride. The larger part of the precipitate was dissolved in hydrochloric acid in slight excess, and boiled with the reserved portion. After filtering, the solution was diluted to definite volume and standardized by precipitating measured portions of the solution with ammonia, filtering on asbestos under pressure in a perforated platinum crucible, igniting the residue and weighing as the oxide. The results recorded in section A of the following table were obtained by precipitating definite volumes of the pure solution of beryllium chloride with ammonium phosphate in a platinum dish, dissolving the precipitate in hydrochloric acid in faint excess, and while hot precipitating slowly with dilute ammonia, boiling (while the solution was kept distinctly ammoniacal) until the flocky precipitate was entirely converted to a fine, powdery, semi-crystalline, rapidly subsiding mass. A quarter to a half hour is necessary under the most favorable conditions to cause this conversion. After cooling, the precipitate was filtered off on asbestos under pressure in a perforated platinum crucible, washed carefully with distilled water, dried, ignited and weighed. The filtrate was tested for beryllium by boiling

* Fresenius, Zeitschrift für analyt. Chemie, 1878, 148.

† This Journal, vol. iv, 111.

with ammonia. None was found in these cases, nor in any of the following work. Faint traces of chloride were found in the residues after ignition after dissolving in nitric acid and testing with silver nitrate.

The results are in every case in excess of the theory for the pyrophosphate derived by ignition of the ammonium beryllium phosphate, possibly because the ammonium chloride present may have a tendency to form a salt too rich in ammonium (as was shown to be the case with the magnesium salt), consequently giving too much phosphoric acid in the ignited residue; or, because of inclusion of the chloride and phosphoric acid. It might reasonably be expected that some phosphoric acid may be held since a trace of chloride was found. Either or both of these substances may have been held mechanically, or in combination.

It was found that by boiling for some time the solution of beryllium chloride with microcosmic salt—(6) section B of the table—precipitating in the same manner as when ammonium phosphate was used—that the same sort of powdery mass remained as was obtained by the ammonium phosphate. The residue being tested for sodium according to the method brought out by Kreider and Breckenridge,* showed sodium present to the amount of 0·0062 grams reckoned as sodium phosphate. It may be reasonably supposed that the presence of the sodium was due to one of two causes, inclusion of the soluble phosphate, or to a tendency on the part of the beryllium to form an ammonium† sodium beryllium phosphate or a sodium‡ beryllium phosphate, both of which are known to exist. Long boiling of the precipitates is tedious, and, unless great care is taken, may involve small losses of material; hence if the same results could be obtained with less boiling such treatment would be decidedly advantageous. The results in section C of the table were obtained by adding microcosmic salt to the hot solutions of the chloride, boiling five minutes, cooling, filtering off on an ashless filter—because of the flocky condition of the precipitate—treating as usual before igniting the residue in a platinum crucible. The results compare well with those obtained by long boiling of the precipitated beryllium—although all are in excess of the theory. That ammonium chloride here, as in cases above, has a marked effect in changing the constitution of the phosphate precipitated by microcosmic salt is not readily seen. It is obvious that the presence of an excess of the soluble phosphate is essential to

* This Journal, vol. ii, 263.

† Persoz, Liebig's Annalen, lxv, 174; Atterberg, Bulletin d. l. Soc. chim. d Paris, xxiv, 358.

‡ Scheffer, Liebig's Annalen, cix, 144.

precipitate the beryllium as the double ammonium phosphate from the results recorded in section D of the table, where, after the precipitate of beryllium phosphate had subsided and the supernatant liquid had been poured off, the precipitate dissolved in hydrochloric acid was brought down again at the boiling temperature with ammonia either alone or in presence of ammonium chloride. The results obtained show that the salt approaches the constitution of the tribasic phosphate, when it is precipitated in presence of a faint excess of phosphoric acid, even though ammonium chloride in large amount be present.

TABLE I.

	$Be_2P_2O_7$ corresponding to $BeCl_2$.			$Be_3P_2O_8$ corresponding to $BeCl_2$.				
	Taken. grm.	Found. grm.	Error. grm.	Taken. grm.	Found. grm.	Error. grm.	$(NH_4)_2PO_4$. grm.	NH_4Cl grm.
				A.				
(1)	0·3578	0·3613	0·0035+				2	--
(2)	0·3578	0·3808	0·0230+				2	--
(3)	0·3578	0·3707	0·0129+				2	--
(4)	0·3578	0·3640	0·0062+				2	--
(5)	0·3578	0·3680	0·0102+				2	30
				B.				
							$HNaNH_4PO_4.4H_2O$	
(6)	0·3578	0·3697	0·0119+					
				C.				
(7)	0·3578	0·3618	0·0040+				1·2	--
(8)	0·3578	0·3680	0·0102+				1·2	--
(9)	0·3578	0·3729	0·0151+				1·2	10
(10)	0·3578	0·3631	0·0053+				1·2	60
				D.				
(11)				0·2700	0·2589	0·0111−	0·5	----
(12)				0·2700	0·2989	0 0289+	0·5	−10
(13)				0·2700	0·2936	0·0236+	0·5	5–60
(14)				0·2700	0·2507	0·0193−	0·5	−60

From the work described it is clear that the ammonium beryllium phosphate is not obtained in ideal condition by precipitating a solution of the chloride with ammonium phosphate. Roessler's own results were likewise only approximately correct, as he states. It is also plain that hydrogen sodium ammonium phosphate precipitates the ammonium beryllium phosphate in a condition as nearly ideal as does the ammonium phosphate, while the effect of the ammonium chloride in either case is not marked in producing a phosphate containing ammonia. Of most importance in obtaining the ammonium salt is an excess of the soluble phosphate, for when the amount of the precipitant is reduced to a little more than the theoretical amount the condition of the phosphate coincides almost exactly with the theory for the tribasic phosphate, even though a large

excess of ammonium chloride be present. When there is an abundance of the precipitant the results are all in excess of the theory, which may be accounted for on the supposition that foreign material is included—the chloride of ammonia and the soluble phosphate—to a greater or less extent by the precipitate. The formation of a phosphate of beryllium containing too much ammonia and phosphoric acid, or, in case of the precipitations by microcosmic salt or sodium by the formation of a sodium ammonium beryllium phosphate and sodium beryllium phosphate (known salts) is not definitely proved.

The Ammonium Zinc Phosphate.

Debray,* Bette† and Heintz‡ separately found that ammonium zinc phosphate is formed by boiling a solution of zinc sulphate with ammonium phosphate. This salt was investigated later by A. Guyard (Hugo Tamm),§ who found that if to a solution of a zinc salt of an organic or a mineral acid supersaturated with ammonia until all the zinc oxide is dissolved and made faintly acid with hydrochloric acid, sodium phosphate be added, a flocky precipitate resulted, which on being kept near the boiling point for some seconds was converted to crystalline zinc ammonium phosphate, which filtered readily and was washed free from impurities with the greatest facility. He found that all the zinc in solution was thrown down as the ammonium zinc phosphate, which on ignition yielded the zinc pyrophosphate. With care in handling this process to avoid an excess of the precipitant, and the presence of sodium and potassium salts (on account of the danger of occlusion) the precipitation of the ammonium zinc phosphate, ignition, and weighing as the pyrophosphate made, Guyard believed, an ideal process for the estimation of zinc. Although there was slight solubility of the salt, it made an insignificant loss when the process was handled properly. Acids present, or certain alkalies to any great extent, increased the solubility of the salt so much that the loss became appreciable. Another source of error was to Guyard's mind loss of zinc during the ignition of the zinc ammonium phosphate with the paper on which the precipitate had been collected. Garrigues‖ found, in estimating zinc in a practical way, that this process advocated by Guyard gives in solutions of zinc free from salts of all metals, even alkaline salts—solutions that from previous steps in analysis, however, must have contained ammonium chloride in large amount—as satisfactory results as Guyard claimed for it.

* Comptes Rendus, lix, 40.
† Liebig's Annalen, xv, 129.
‡ Liebig's Annalen, cxliii, 156.
§ Chemical News, xxiv, 148.
‖ Jour. Am. Chem. Soc., xix, 936.

Garrigues' method of procedure was to add acid diammonium phosphate to a warm solution of zinc exactly neutralized with either hydrochloric acid or ammonia so that the weights of zinc ammonium phosphate and that of the diammonium phosphate added should be as one to five respectively, to heat until the flocky precipitate becomes crystalline and subsides, filtering off on asbestos, drying at 100° C. and weighing preferably, although the residue may be ignited without loss, since the filtration is made on asbestos in a perforated crucible. Langmuir* modifies the method by destroying with dilute acetic acid any free ammonia that may be left in the solution after boiling.

In the work that follows, in which an attempt was made to show what precipitate is formed from a solution of zinc by the action of a soluble phosphate, also what effect ammonium chloride has upon the precipitate, a solution of zinc chloride prepared as detailed below was employed. The pure zinc chloride of commerce was treated with zinc carbonate, filtered and precipitated with ammonium sulphide. This precipitate was boiled in a slight excess of hydrochloric acid until all the hydrogen sulphide was removed, and then was precipitated with sodium carbonate. After washing carefully until all the chloride was removed, the greater part of the carbonate was dissolved in sulphuric acid in slight excess, boiled with the remaining portion of the carbonate and filtered. This solution diluted to definite volume was standardized as sulphate by evaporating the solution to dryness in a platinum crucible and heating the residue.† The heating is carried on safely by so placing the platinum crucible in a radiator (consisting of a crucible and a triangle) that the bottom of the platinum crucible was held about one centimeter above the bottom of the outside crucible. Constant weights were obtained in successive treatment with a few drops of sulphuric acid and heating over the radiator. The results obtained in this manner were a trifle higher, though in fair agreement (when the nature of the carbonate process is taken into consideration) with determinations of the zinc in the solutions as oxide after precipitating with sodium carbonate with the usual precautions, filtering off on asbestos under pressure in a perforated platinum crucible, washing with distilled water, drying and igniting. Results are given in Table II showing the amount of zinc sulphate found in five different portions each of forty cubic centimeters of the solution of zinc sulphate, and, for comparison, the results of determinations as zinc oxide by the carbonate processes are included.

* Jour. Am. Chem. Soc, xxi, 115.

† Rose-Finkener Analytische Chemie, 6te Auflage, vol. ii, 117.

TABLE II.

$ZnSO_4$ found in 40^{cm^3} of solution. grm.	Mean value of ZnO corresponding to $ZnSO_4$ in 40^{cm^3} of solution. grm.	ZnO found in 40^{cm^3} of solution by precipitation as the carbonate. grm.
0·5386	0·2712	0·2691
0·5385		0.2685
0·5387		0·2711
0·5387		
0·5390		

Definite portions of the solution of zinc sulphate were carefully drawn from a burette into a platinum dish, heated and treated with ammonium phosphate until the solution turned red litmus paper blue. The whole was heated until the flocky precipitate became crystalline and fell to the bottom of the dish. The solution after standing as recorded in section A of the table was filtered off on asbestos under pressure in a perforated platinum crucible, and the precipitate was washed with distilled water, dried, ignited and weighed. The filtrate in each case, as in all following cases, was tested for zinc with sulphuretted hydrogen. The results recorded in section B of the table were obtained in the same manner as those of section A, with microcosmic salt substituted for the ammonium salt as the precipitant. The results are below the theory for the pyrophosphate, but no appreciable amount of zinc appeared in the filtrates. Neither ammonium phosphate nor ammonium sodium phosphate seems to precipitate the ideal ammonium zinc phosphate under these conditions; and the time of standing appears to be without effect.

The results recorded in section C were obtained by precipitating the warm solution of the zinc in presence of large amounts of ammonium chloride by adding microcosmic salt until the solution was alkaline to litmus. From these results it seems that the presence of ammonium chloride is essential for the conversion of the zinc phosphate precipitated by hydrogen sodium ammonium phosphate to the ammonium zinc salt. As a matter of fact the solutions employed by Guyard and those in which estimations are made by practical workers do contain ammonium chloride formed in previous steps of the analysis. The proportion of zinc to phosphate suggested by Garrigues—1 : 5—is the amount of soluble phosphate necessary to turn red litmus blue after the zinc is precipitated. In order to find out whether the presence of so large an amount of the soluble phosphate is necessary in presence of ammonium chloride, the solution of zinc sulphate was precipitated in presence of the necessary amount of ammonium chloride by the microcosmic salt, in small excess above the equivalent of

the ammonium phosphate, and the solution was made just ammoniacal to litmus with a few drops of dilute ammonia both before and after heating to convert it to crystalline condition. Experiment (15) shows that precipitation is not complete under these conditions. The zinc left in the solution was precipitated at once as sulphide, and estimated as the oxide, after dissolving in hydrochloric acid and precipitating with sodium carbonate. In (16) of the table the first filtrate was treated with an excess of microcosmic salt, and boiled. Another portion of the ammonium zinc phosphate was precipitated, and was filtered off and estimated. No zinc was found by sulphuretted hydrogen in the second filtrate.

TABLE III.

	$Zn_2P_2O_7$ corresponding to $ZnSO_4$. Taken. grm.	Found. grm.	Error. grm.	Error in terms of Zinc. grm.	$Zn_2P_2O_7$ corresponding to Zn left in the filtrate. grm.	$(NH_4)_2PO_4$. grm.	NH_4Cl. grm.	Time of standing. hours.
				A				
(1)	0·6355	0·6206	0·0149—	0·0060—	trace	3 13	----	1½
(2)	0·6355	0·6254	0·0101—	0·0040—	trace	3·13	----	16
(3)	0·6355	0·6300	0·0055—	0·0022—	trace	3·13	----	16
				B		$HNaNH_4PO_4.4H_2O$ grm.		
(4)	0 6355	0·6271	0·0084—	0·0034—	trace	4·47	0·5	1
(5)	0 6355	0·6256	0·0099—	0·0040—	none	4·47	0·5	20
				C				
(6)	0·6355	0·6285	0·0070—	0·0028—	none	4·47	10	½
(7)	0·6355	0·6304	0·0051—	0·0020—	none	4·47	10	¾
(8)	0·6355	0·6295	0·0060—	0·0024—	none	4·47	10	2½
(9)	0·6355	0·6335	0·0020—	0·0008—	none	4·47	10	16
(10)	0·6355	0·6381	0·0026+	0·0010+	none	4·47	20	½
(11)	0·6355	0·6379	0·0024+	0·0009+	none	4·47	20	2
(12)	0·6355	0·6386	0·0031+	0·0012+	none	4·47	20	½
(13)	0·6355	0·6393	0·0038+	0·0014+	none	4·47	20	½
(14)	0·6367	0·6355	0·0012+	0·0005+	none	4·47	30	16
				D				
(15)	0·6355	0·6172	0·0183—	0·0072—	0·0108	0·894	20	3
(16)	0·6355	I 0·6227 II 0·0040	0·0098—	0·0039—	none	I 0·894 II 3·576	20	½
				E				
(17)	0·6355	0·6270	0·0085—	0·0034—	none	4·47	----	3
(18)	0·6355	0 6125	0·0230—	0·0093—	0·0148	4·47	----	18
(19)	0·6355	0·6303	0·0052—	0·0021—	0·0020	4·47	10	18

From the results it seems obvious also that an excess of the soluble phosphate is necessary to complete the precipitation of the zinc as the ammonium zinc phosphate instead of partly ammonium zinc phosphate and partly tribasic phosphate.

In section E of the table are recorded results where the precipitation was made in presence of an excess of the precipitant either alone or in presence of ammonium chloride, the solution being made faintly acid to litmus with acetic acid, according to the manner in which Langmuir recommends to conduct the precipitation. All the results by the method are low. The condition of the ammonium zinc phosphate most nearly approximating to the ideal is obtained as shown in (9) to (14) by precipitating in presence of ammonium chloride in large amount. Microcosmic salt is added until the solution containing the ammonium salt is alkaline and the whole is heated until the mass subsides in crystalline condition. The amount of ammonium chloride should be twenty grams if the filtration is to be made as soon as the solution cools. One-half the amount will do if the liquid stands a number of hours. Larger amounts tend to give a salt too rich in ammonia. The time of standing seems to be a less important factor than either the excess of microcosmic salt or ammonium chloride.

The Ammonium Cadmium Phosphate.

According to S. Drewsen* the cadmium ammonium phosphate is precipitated by allowing a solution of cadmium sulphate to stand twenty-four hours with ammonium phosphate. It is very soluble both in acids and alkalies. No further preparation of this seems to have been recorded. For the work on this salt to be given below, made with reference to determining the constitution of the salt by hydrogen sodium ammonium phosphate, the effect of ammonium chloride in the precipitation, and the value of the salt for quantitative work, the solution of cadmium chloride employed was prepared as follows: A solution of cadmium sulphate acidulated with hydrochloric acid was precipitated with sulphuretted hydrogen, filtered and washed, and the precipitated sulphide was dissolved in hydrochloric acid and filtered from possible traces of copper and lead. The solution of the sulphide in hydrochloric acid was boiled until all the sulphuretted hydrogen was expelled, and filtered on asbestos in a perforated crucible of platinum under pressure. The cadmium in the filtrate precipitated with ammonium carbonate in excess was washed free from chloride, dissolved in hydrochloric acid and diluted to definite volume. It was standardized as oxide† after precipitating with sodium carbonate with the necessary precautions.

The standard solution of cadmium chloride was drawn carefully from a burette into a platinum dish, and, while hot, was precipitated by adding hydrogen sodium ammonium phosphate

* Gmelin-Kraut, 6te Auflage, iii, 74.
† Browning, this Journal, xlvi, 280.

until the solution was alkaline to litmus. After heating until the solution became crystalline, the whole stood three hours in case of (1) of the table and sixteen hours in case of (2) and (3), before filtering. In experiments (4) to (12), inclusive, recorded in the table, precipitation was made in the same manner as in (1) to (3) in presence of varying amounts of ammonium chloride, and the precipitates were filtered after standing as stated below in the table. It is clear from the results that the cadmium separates out completely on long standing only. Moreover, the ideal condition of the ammonium cadmium phosphate is obtained only when an abundance of ammonium chloride is present; but large amounts of ammonium chloride

TABLE IV.

	$Cd_2P_2O_7$ corresponding to $CdCl_2$. Taken. grm.	Found. grm.	Error. grm.	Error in terms of Cadmium. grm.	$Cd_2P_2O_7$ corresponding to Cd found in the filtrate. grm.	$HNaNH_4PO_4 \cdot 4H_2O$. grm.	NH_4Cl. grm.	Time of standing. hrs.
(1)	0·6972	0·6201	0·0771—	0·0434—	0·0059	4·5	----	3
(2)	0·6972	0·6135	0·0837—	0·0471—	none	4·5	----	16
(3)	0·6972	0·6134	0·0838—	0·0471—	none	4·5	----	16
(4)	0·6972	0·6792	0·0180—	0·0101--	trace	4·5	1	16
(5)	0·6972	0·6831	0·0141—	0·0079—	0·0113	4·5	10	2
(6)	0·6972	0·6976	0·0004+	0·0002+	trace	4·5	10	16
(7)	0·6972	0·6969	0·0003—	0·0002—	trace	4·5	10	18
(8)	0·6972	0·6962	0·0010—	0·0006—	trace	4·5	10	16
(9)	0·6972	0·6891	0·0081—	0·0045—	0·0191	4·5	20	16
(10)	0·6972	0·6972	0·0000	0·0000	trace	4·5	20	16
(11)	0·6972	0·6942	0·0030—	0·0016—	trace	4·5	20	16
(12)	0·6972	0·6737	0·0235—	0·0132—	0·0304	4·5	30	16
(13)	0·6972	0·5655	0·1317—	0·0741—	0·1378	4·5	30	16
(14)	0·6972	0·6922	0·0050—	0·0023—	0·0088	4·5	10	16
(15)	0·6972	0·3209	0·3763—	0·2117—	0·2449	4·5	----	16

dissolve this salt. In (14), where ammonia was added after precipitation was complete, the salt dissolved somewhat; also in (15), where the solution was left faintly acid with acetic acid, a large part of the salt was dissolved. These weights of cadmium dissolved in the filtrate were obtained by treating the filtrates with sulphuretted hydrogen, dissolving the sulphide in nitric acid, and weighing as oxide after precipitating with sodium carbonate.

The ammonium cadmium phosphate is obtained in ideal condition by precipitating with microcosmic salt in presence of 10 grms. ammonium chloride in a total volume of 100^{cm} to 150^{cm}—shown in (6), (7) and (8)—filtering after standing some time. On drying and igniting the pyrophosphate is left. Very large amounts of ammonium chloride—30 grms.—dissolve the salt, and seem to tend to cause the formation of a

phosphate too rich in ammonia. Either acid or ammonia in small amount dissolves the salt, as is shown in (14) and (15).

The results of this investigation as to the analytical application of the double ammonium phosphates of beryllium, zinc, and cadmium may be summarized briefly as follows: It is impossible to estimate beryllium with accuracy as the pyrophosphate obtained by igniting the double ammonium phosphate precipitated from beryllium solutions by microcosmic salt or ammonium phosphate in presence of ammonium chloride. In presence of the proper amount of ammonium chloride (10 grm. to 20 grm. in 100^{cm^3}–200^{cm^3} of liquid) zinc ammonium phosphate can be obtained in the ideal condition, which on ignition yields the pyrophosphate. This method may serve, therefore, for the accurate estimation of zinc.

Cadmium may be estimated with accuracy as the pyrophosphate if the precipitate by microcosmic salt in the nearly neutral solution containing ammonium chloride in the proportion of ten grams to one hundred cubic centimeters is allowed to stand several hours before filtering. In this way all cadmium separates out from the solution as a beautiful crystalline mass of cadmium ammonium phosphate of ideal constitution. The conditions must, however, be preserved with care; there must be no excess of ammonia, no free acid, and no excess of ammonium salt beyond the quantity indicated, while that amount is necessary.

ART. XXII.—*Separation of Iron from Chromium, Zirconium, and Beryllium, by the Action of Gaseous Hydrochloric Acid on the Oxides;* by FRANKE STUART HAVENS and ARTHUR FITCH WAY.

[Contributions from Kent Chemical Laboratory of Yale University—LXXXVII.]

IT has been shown in a former paper from this laboratory* that iron oxide may be completely volatilized as chloride by a strong current of hydrochloric acid gas acting at a temperature of 450–500°, and also that the addition of a little free chlorine to the gaseous hydrochloric acid renders this action complete at lower temperatures, 180–200°, without the danger of error arising from the liability of ferric chloride to dissociation, or from deficiency of oxidation in the oxide treated, or mechanical loss due to too rapid volatilization. It has also been shown that this reaction can be employed for the separation of iron and aluminum, taken as the oxides, and its application to the separation of iron from other metallic oxides has been suggested.

The oxides of chromium, zirconium, and beryllium, like aluminum oxide, are not acted upon by a current of dry hydrochloric acid gas at the temperatures before mentioned, and these oxides also can be entirely freed from iron by this reaction, as the experiments to be described will show. The procedure was the same in each case and analogous to that employed for the separation of iron from aluminum. A mixture of a weighed portion of one of these oxides with a weighed portion of ferric oxide, contained in a porcelain boat and placed within a roomy glass tube supported in a small combustion furnace, was submitted to the action of a dry current of hydrochloric acid gas and chlorine generated by dropping sulphuric acid upon a mixture of strong hydrochloric acid, common salt, and a small amount of manganese dioxide. The gas was admitted at one end of the combustion tube and passed out at the other through a water trap, while the required temperature, from 200°–300°, was maintained by regulating the various burners of the furnace. The time of action varies somewhat with the condition of the oxide to be volatilized, and the temperature, generally an hour's heating at 200°, proves sufficient for the complete removal of 0·1 gram of iron. At higher temperatures the action is more rapid; but the lighter oxide, the beryllium especially, is liable to mechanical loss through the too rapid volatilization of the iron, as experiment (17), where a temperature of 500° was used, will show. It is

* Gooch and Havens, this Journal,

better, therefore, to use lower temperatures, raising the heat for a few minutes when the action is apparently complete to ensure the removal of the last traces of iron. Tests showed the residual oxides from which the ferric oxide had been removed in this manner to be entirely free from iron.

Exp.	Fe_2O_3 taken. grams.	Cr_2O_3 taken. grams.	Cr_2O_3 found. grams.	Error. grams.
1	----	0·1008	0·1008	0·0000
2	0·1007	0·1006	0·1006	0·0000
3	0·1007	0·1000	0·1002	+0·0002
4	0·1010	0·1005	0·1003	−0·0002
5	0·1019	0·1006	0·1005	−0·0001
6	0·2007	0·1003	0·0999	−0·0004
		ZrO_2 taken. grams.	ZrO_2 found. grams.	
7	----	0·1516	0·1516	0·0000
8	0·1053	0·1010	0·1010	0·0000
9	0·1204	0·1519	0·1523	+0·0004
10	0·1236	0·1516	0·1517	+0·0001
11	0·2150	0·1517	0·1519	+0·0002
		BeO taken. grams.	BeO found. grams.	
12	----	0·1309	0·1311	+0·0002
13	----	0·1285	0·1285	0·0000
14	0·0997	0·0456	0·0457	+0·0001
15	0·1045	0·1099	0·1099	0·0000
16	0·1215	0·1080	0·1081	+0·0001
17	0·1510	0·1305	0·1290	−0·0015
18	0·2030	0·1081	0·1083	+0·0002

The separation of iron from chromium, zirconium, and beryllium by this method is obviously complete within very satisfactory limits of error.

ART. XXIII.—*An Albertite-like Asphalt in the Choctaw Nation, Indian Territory;** by JOSEPH A. TAFF.

History of its development.

IN the fall of 1897, while surveying the southern part of the McAlister Quadrangle in the Indian Territory, coal-like stringers interstratified with greenish shale were discovered in McGee Creek Valley. The stringers were one foot and less in thickness, and in places were intermingled with fragments of shale of the same nature as that in the wall rock. The shale upon both sides was of the same kind, was not carbonaceous, and was not of the nature of fire-clay as is usually found in contact with coal. The relation between the carbonaceous substance and the shale did not attract especial comment, since the rocks were nearly on edge and supposed to be in a faulted zone.

On working farther south in Impson Valley, T. 1 S., R. 15 E., an exposure of the same coal-like mineral was found cropping out in Ten Mile Creek in the same formation as on McGee Creek. The bed was partially concealed by water, but by good authority it was reported to be four feet thick. Still farther south on the west side of Impson valley, near the south side of T. 1 S., R, 15 E., the same mineral had been prospected and exploited on a small scale. A shaft twenty feet in depth had been sunk upon its outcrop where it was reported to be 25 feet thick and almost upon edge. The shaft was full of water at the time and the coal concealed.

The deposit was discovered by prospectors in 1890, exploited in 1892 and a little later abandoned because of the steep dip and friability of the product. Specimens were collected by the writer and submitted with a series of bituminous coals from the McAlister region for proximate analysis. These analyses were made by Dr. W. F. Hillebrand, and are placed here for comparison.

	Water.	Volatile Combustible Material.	Fixed Carbon.	Ash.	Sulphur.	Phosphorus.
Albertite.	·25	42·33	55·97	1·45	1·47	0·00
Coal. (Hartshorne)	1·63	38·27	55·15	4·84	1·51	0·014

The low percentages of water and ash in the albertite are the only features that would attract attention. On applying a flame to a fragment of the mineral it softens, disintegrates and ignites, burning for a short time after the removal of the flame. This, however, connected with the fusibility and friability of the mineral, indicates strongly that it is not a true coal.

* Published by permission of the Director of the U. S. Geological Survey.

On returning to the field in 1898, a second inspection of the three localities of the mineral was made. It was found that Mr. Geo. D. Moulton had leased the lands including the prospects in Impson Valley. The mine near the south side of T. 1 S., R. 15 E., was being operated. Entries were made along the strike for 100 feet to both the foot and hanging walls. The mineral was found to form a vein, cutting folded green clay shale and striking nearly north and south and approximately parallel with the general trend of the rocks. The vein dips about 70 degrees east. The shale at both foot and hanging walls has been crushed so that its structure is destroyed and slickensided surfaces extensively developed. Two sets of joint planes bearing respectively northeast and northwest intersect the vein. The vein material, also, in some of the joints is striated, showing that there has been movement along such faces since the solidification of the mass.

The development of systems of joints in the vein intersecting approximately at 45 degrees to the face of the wall would indicate crushing pressure perpendicular to such faces while the mineral was in a solid or semi-solid state. The whole of the vein at the time when excavations had been made in it to a depth of about 30 feet showed indications of apparent crushing.

Mr. Moulton, the owner and operator of the mine, reports that at a depth of about 35 feet the vein for 12 feet inward from the foot wall has changed in structure. The specimens submitted by him, though small, show a compact mass with a smooth conchoidal fracture in contrast to the friable product with hackly surface found in the upper part of the vein and in the entire body of the vein to a depth of more than 30 feet.

Samples were collected from the face of the working 30 feet beneath the surface and submitted to Dr. Wm. C. Day for complete analysis. I quote the essential part of Dr. Day's report of the analysis. Duplicate tests were made in the determination of specific gravity, of solubility, and in the chemical analysis.

"*Report on the hydrocarbonaceous material supplied by Mr. Joseph A. Taff of the U. S. Geological Survey.*

The material is extremely brittle, pulverizing almost at a touch; it takes fire readily. It shows no sign of conchoidal fracture. Heated in a tube it does not melt but softens and gradually cokes progressively without becoming liquid at any one time.

Distillation of the asphalt from a retort showed that it does not entirely melt at any one time but softens and decomposes

simultaneously like a coal. The distillate is dark red by transmitted light and shows a decided green fluorescence. For sake of comparison a quantity of soft coal used for making coal gas and obtained from a gas works was distilled. This gave a more tarry distillate but less in amount for equal weights of material. The odor of the distillate from the coal was markedly different from that from the asphalt; it lacked also the green fluorescence of the distillate from the asphalt. The coal was practically insoluble in carbon disulphide, while the asphalt was soluble to the extent of over 35 per cent.

The material, all things considered, resembles albertite more than any other of the asphalt substances, but it differs from it too in certain respects. For the sake of comparison the data contained in Dana's Mineralogy on albertite are placed here side by side with the corresponding data pertaining to the material supplied by Mr. Taff.

Albertite.	Mr. Taff's material.
Specific gravity 1·097.*	Specific gravity 1·175.
Color, jet black.	Color, jet black.
Softens a little in boiling water.	Does not soften in boiling water.
In the flame of a candle shows incipient fusion.	In flame of candle shows incipient fusion and takes fire, burning for a short time after removing from the flame.
Only a trace soluble in alcohol.	Only a trace soluble in alcohol.
4% soluble in ether.	5·34% soluble in ether.
30% soluble in turpentine.	Almost insoluble in turpentine.

Per cent. of carbon as found by Wetherill	86·04	Per cent. carbon	86·57
Per cent. hydrogen (Wetherill)	8·96	" hydrogen	7·26
" nitrogen (Wetherill)	2·93		
" nitrogen (Day)	1·84	" nitrogen	1·48
" sulphur (Wetherill)	trace		
" sulphur (Day)	0·17	" sulphur	1·38
" oxygen (Wetherill)	1·97	" oxygen	2·00
" ash (Wetherill)	0·10	" ash	1·31

Of the differences between albertite and the Taff asphalt the contrast in the solubility in turpentine is perhaps the most striking.

While the material submitted by Mr. Taff resembles a coal in the way it softens and decomposes under the influence of

* I find for Nova Scotia albertite sp. gr. 1·074 at 20·9° C.

heat instead of entirely melting as asphalts generally do, the distillation products are markedly different from those of coals in abundance, in fluorescence, and in odor.

As the result of examining the analytical figures given in Dana's Mineralogy for a long list of coals, I can find none which even approximate those found for the material in question. The solubility in carbon disulphide classes the material with the asphalts, rather than with the coals. The analytical figures and the solubility show that the material resembles albertite from Nova Scotia more than any other of the asphalt-like bodies which have thus far been investigated."

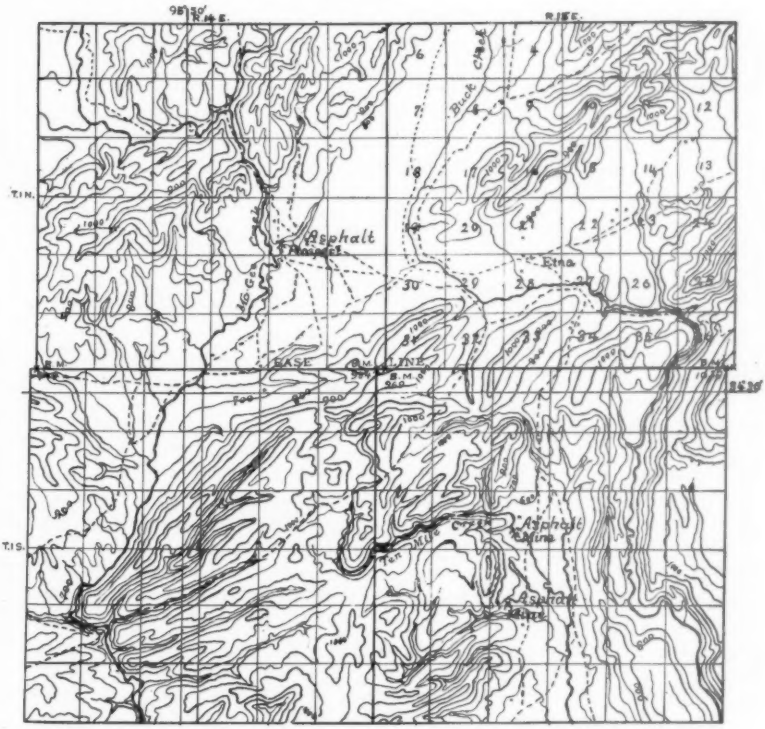
Geology of Associated Rocks.

The map will show the general topographic character of the country as well as the located occurrences of albertite. It also gives the township land surveys which substitutes a scale for any location. The mapped district lies in both the McAlister and the Antlers Quadrangle. The parallel of latitude, 34° 30′, is the line between them, the McAlister being on the north. The contours mark elevations above the sea level and are at intervals of 100 feet.

The topography of the country is characteristic of the Ouachita mountain system in the Indian Territory to which it belongs. It is a system of rugged and generally parallel stony hills and small mountains which rise to elevations of from a few hundred to a thousand and even more feet above nearly level plain-like valleys which are often several miles wide and many miles long. These plain-like valleys are governed by and limited to the outcrop of shales and other associated friable rocks. By observing the map a wide valley may be noted extending from McGee Creek in the north part of T. 1 S., R. 14 E. northwestward to Buck Creek, where it is divided by the Jack Fork Mountains. There is another such valley known locally as Impson Valley in the center T. 1 S., R. 15 E., in which the asphalt mines are located. The mountains, hills and all elevated lands are rugged and are held up by many beds of hard sandstone whose edges are exposed. The streams in the plains have little or no valleys of their own. Their channels, also, are little more than ditches cut in the soil and soft rock. The streams which flow in or across the hilly country are confined to narrow and often deep gorges or valleys.

The structure of the rocks, especially that of the shales, is hard to interpret. Besides having been folded and faulted to an extent difficult of appreciation, the beds are almost entirely concealed by soil and rock debris. It has been clearly determined, however, that the beds have been folded along lines

northeast and southwest, then overturned towards the northwest and, in many places, faulted. Especially where shales are involved the crushing and faulting has been very extensive. Three lines of faulting bearing northeast and southwest and having displacements of many hundred feet, extend along the southeast sides of the group of hills through the center of T. 1 N., R. 14 E., of the Jack Fork Hills in the center of T. 1 N., R. 15 E. and of the hill in section 3, T. 1 N., R. 15 E. respectively. The latter two lines of faulting extend from the ends of the hills southwestward into the plain toward the albertite exposures.



Map to show location of asphalt mines in Choctaw Nation.

Another zone of faulting extends from the plain nearly two miles east of Etna bearing southwestward, crossing the center of the south side of T. 1 N., R. 15 E. thence nearly due south along the west side of Impson Valley. The albertite mines of this valley are located along this fractured zone. The north

end of this valley, in Section 10, T. 1 S., R. 15 E. is anticlinal and the rocks along the east side of the valley dip normally toward the east. On the west side, however, the older shales which contain the albertite are thrust upward against the younger sandstones of the hills.

The geology, were it not for the complications of structure, would be simple, For all purposes necessary here, however, the problem of the geology may be briefly stated. The shales which lie in the plains and wide valleys noted above, and which contain the albertite, are the oldest known in this district. Their thickness is difficult to determine, though it is estimated to be several thousand feet. They are composed of greenish and bluish clay shales, sandy shale and greenish friable sandstone. Thin beds of black siliceous slate have been noted in the upper part of this body of shale.

All the thick sandstones which form the hills and ridges lie above the shales. The aggregate thickness of these sandstones is estimated also to be several thousand feet. Beds of limestone which have been found to occur just above the sandstone formation contain fossils of Ordovician age. Hence it is concluded that the sandstones and the shales which contain the albertite are of Ordovician age or older.

Conclusions.

The mineral, in both its physical and chemical properties, is shown to be an asphalt and more closely related to albertite than to any other known asphalt substance. Indeed its only variation from albertite of any moment is its solubility in turpentine. The friability and fracture of the sample submitted for analysis is, without much doubt, due to its structural condition produced by pressure after solidification. The product occurs as veins or dikes filling fissures along zones of thrust faulting. It occurs in shales of, at present, unknown age.

The quantity of this asphalt can be conjectured only. As far as known it occurs in thin stringers, in a vein about 4 feet thick, and in a vein about 25 feet thick. The surface of the shale containing the product is worn down in valleys and concealed by soil or overwash so that the asphalt is known only so far as prospected. From the manner of its occurrence in fissures produced by fracture, where it has suffered probably great pressure, the vein is supposed to vary in thickness and in longitudinal extent and may descend to any reasonable depth.

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ART. XXIV.—*Notice of a new Meteorite from Murphy, Cherokee Co., N. C.;* by HENRY L. WARD, Rochester, N. Y. With Plate IV.

IN May of the present year we received word from Mr. W. B. Lenoir that he had what he supposed to be a meteorite that he desired to sell. Upon request he forwarded it to us; and a superficial glance at the well-pitted surface was sufficient to determine that it was a siderite.

Under date of May 25th, Mr. Lenoir writes: "It was ploughed up or washed out in a field in Cherokee Co., N. C., five miles from Murphy. From the location [where] found I think it must have been washed out by some of the immense rains which fell in that section last winter. . . . The small piece enclosed with the larger one was broken off when I purchased it. . . . The stone [iron] I understand was found about six weeks ago." Later he writes: "In answer to yours of June 10, asking how the meteorite was broken, will say that I did not break the meteorite. The man who first showed me the piece said that they attempted to cut it with a cold chisel and did not succeed. By some means (I think he said) they cut around it and then either with a blow or by prizing broke it off. . . . He said he had a 'hell of a time' breaking it."

As was to be expected, this meteorite adds another to the great majority of siderites the date of whose fall is unknown.

Fig. 1, Plate IV, shows the iron with the broken piece set in place so as to give the original form. Its height is $23{\cdot}5^{cm}$. A rectangle drawn about the base upon which it stands measures $13{\cdot}4 \times 11{\cdot}5^{cm}$. The weight of the iron was 7753 grams (17 lbs. $1\frac{1}{2}$ oz.), of which the broken end weighed 808 grams. Another photograph of the entire iron was taken to show the remarkably sharp angle formed by the left hand edge and the side opposite to that shown in fig. 1; but a defect in the plate rendered this photograph unusable. Fig. 2, showing end views of both pieces from the broken surface, gives something of this angularity, which was rather more marked lower down. In some of the larger sections cut across the iron this angle is rather more acute than a right angle, as this side is somewhat concave, and the edge is very sharp. These two surfaces are less deeply pitted than the one shown in the figure; and convey the impression that the meteorite in hand is but a fragment of a larger one that broke not far above the earth. Had it traveled far since dividing we would expect the angles to be rounded. An examination of the crust fails to give any evidence for or against this theory. Flow lines, if they ever

existed, have been removed by weathering and the oxidized crust appears equally thin on all sides. The fact that the edges of the mass are approximately parallel to the lines of crystallization is only in accordance with what has been shown to be common to siderites in general.

The square fracture is an interesting feature that I believe to be quite unusual in iron meteorites. Its surface is nearly a parallelogram $4{\cdot}8 \times 3{\cdot}3^{cm}$, with one of the shorter sides surmounted by a triangle $3{\cdot}5^{cm}$ in height; giving a surface of approximately $21{\cdot}5^{sq\ cm}$. One side shows that it has been cut by a cold chisel to a depth of about 2^{mm}. The rest of the face is a clean straight break with a hackly surface. This is shown on the larger mass in fig. 2.

The etched surface presents two main series of lines enclosing rhombs having the angles 161·2 and 18·8; other lines crossing these produce all the figures compatible with the twinning about a cube with the exception of a single line, which probably exists but which I have been unable to find.

Under the microscope each higher power up to about one hundred diameters reveals lines not seen with lower powers. That the series of lines giving the angles above mentioned are the primary ones, is indicated by the fact that the lines of fracture on the broken face have followed these. On a section cut at right angles to these, the lines apparent to the eye give decidedly larger figures; but under the microscope this distinction disappears.

The Neumann lines were visible in the photograph of the etched end shown in fig. 2; but are entirely lost by the screen used in making the half tone; consequently we introduce a print directly from an etched section of the iron, fig. 3, which under a hand lense should show the lines fairly well.

Troilites appear in all the sections; but are in most cases of very small size. The largest one that appears measures 9×13^{cm} in diameter. It is interesting to note that the Neumann lines are materially flexed immediately about some of the troilites: indicating that they existed in a plastic condition during the growth of the troilites.

Daubreelite, in unusually large masses, occurs in two of the slices; both as veins crossing the troilites and as solid masses at the sides. The largest mass measures, on its two nearly rectangular faces, 5·5 and 5^{mm}; the other sides being formed by a segment of the nearly circular border of the troilite within which it has formed.

An analysis of the iron has not yet been made.

Unfortunately the more euphonious title "Cherokee County" has already been applied to the Losttown, Cherokee Co., Ga., siderite, and we are forced to adopt the less pleasing one of the town near which it was found, *Murphy*, as the name for this meteorite.

ART. XXV.—*On the Separation of Alumina from Molten Magmas, and the formation of Corundum;* by J. H. PRATT.

SOME interesting problems have arisen, in recent years, regarding the differentiation of igneous magmas upon cooling. It has been the author's privilege during the last three years to study in the field various occurrences of corundum, spinel and chromite in peridotite rocks, and in some recent papers* the igneous origin and the chemical composition of these minerals have been discussed by him. It is the intention in this paper to give those field observations that have a theoretical bearing upon the separation of these minerals from a molten magma and more especially of the alumina.

The results of the elaborate studies that have been made by Vogt,† Morozewicz,‡ Lagorio§ and others upon the artificial formation of minerals in molten magmas, have been verified by observations upon the occurrence of the same minerals in nature that have also separated out from molten magmas.

The separation of alumina is well illustrated in nature in the occurrence of corundum, spinel and chromite in the rocks of the peridotite group. Of these rocks, the most common one containing these three minerals is dunite, consisting almost entirely of chrysolite or its alteration product, serpentine. In the analyses of the dunite rocks, they show either a trace or very small percentage of alumina or chromic oxide or even none; and in the analyses of other peridotites than dunite, the same usually holds true for amounts beyond those required in the composition of the normal minerals that compose these rocks.

The peridotite localities that have been examined by the author are all located in the western part of North Carolina, and at these various localities the following phenomena have been observed:

1. Peridotite rocks containing small particles and grains of chromite but no corundum or spinel.

2. Peridotite rocks containing deposits of chromite (which upon analysis showed a variable per cent of alumina and magnesia), but no corundum or spinel.

3. Peridotite rocks containing chromite, corundum and spinel.

* This Journal, vol. vi, July, 1898, p. 49; ibid. vol. vii, April, 1899, p. 281, and Trans. Am. Inst. Mining Eng., vol. xxix, 1899.

† Zeitschr. für Prakt. Geol., Nos. 1, 4 and 7, 1893.

‡ Zeitschr. für Kryst., vol. xxiv, p. 281, 1895, and Tschermak's Min. u. Petrog. Mittheilungen, Bd. xviii. H. 1–2–3, pp. 1–90 and 105–240, 1898.

§ Zeitschr. für Kryst., vol. xxiv, p. 285, 1895.

4. Peridotite rocks containing chromite and corundum, but no spinel.

5. Peridotite rocks containing corundum and feldspar.

1 *and* 2. In the analyses of the chromites that have come under the author's notice, all but two show the presence of alumina and magnesia in varying amounts. In some of them the percentage of alumina and magnesia is very large and where in the ordinary chromite the formula may be represented by 9 ($FeO.Cr_2O_3$), ($MgO.Cr_2O_3$), 2 ($MgO.Al_2O_3$); at one locality near Webster, Jackson County, a chromite (*mitchellite*), was found which had the formula ($FeO.Cr_2O_3$), ($MgO.Cr_2O_3$), 2 ($MgO.Al_2O_3$). Thus when there is considerable chromite found in these rocks, there is usually no, or at most but a trace of corundum present, and from what has been said above regarding the analyses of chromite, it would seem to be the case that when there is but little alumina and only a small excess of magnesia in peridotite magmas, containing considerable chromic oxide, that they unite to form the spinel molecule which instead of separating out independently as the mineral spinel, enters into combination with the $FeO.Cr_2O_3$ molecule in the mineral chromite.

3. At a few of the peridotite localities both corundum and spinel have been found, as at the Corundum Hill Mine, where a fine-grained, almost black spinel occurs through which is scattered particles and masses of corundum; and at the Carter Mine near Democrat, where there is a great abundance of corundum and a green black spinel. In peridotite magmas of this type, it would seem that there has been an excess of magnesia present which has united with a portion of the alumina present to form the molecule $MgO.Al_2O_3$, which separated out as spinel. The remaining portion of the alumina formed corundum.

Undoubtedly there is a strong affinity between magnesia and alumina which tends to form the spinel molecule; but in these peridotite magmas, it is only the excess of magnesia over that required for the normal magnesian silicates that has united with the alumina. Except alteration products there are no alumina-magnesia silicates found in these rocks, showing that in magmas of this type there is no tendency for the alumina to unite with the magnesia in forming double silicates.

4. At most of the peridotite localities there has been no spinel at all observed, but there is often a considerable quantity of corundum found. The chromite at all these localities shows the presence of a certain percentage of alumina and magnesia. Thus the small excess of magnesia unites with a definite amount of the alumina, but instead of separating out as spinel, it separates out in the chromite molecule as previ-

ously mentioned. The greater part of the alumina separates out as corundum.

5. In a number of these peridotite formations feldspar has been found which is undoubtedly one of the original minerals of the rock and not a secondary product.

At the Cullakeenee Mine, Buck Creek, Clay County, there is, at one of the contact corundum veins, a large amount of feldspar and hornblende which have separated out with the corundum: again at the Bad Creek Mine, Sapphire, Jackson County, the corundum is found associated with feldspar and biotite mica. In both places they are lime-soda feldspars, and this occurrence of feldspar and corundum in a peridotite rock indicates that the molten magma contained some of the alkali and alkali earth oxides, as Na_2O, K_2O and CaO, and that a portion of the alumina united in the formation of the feldspar molecule and that the rest of it separated out as corundum. In a magma of this type, there is present a large amount of magnesia which forms the magnesian silicates but apparently has no tendency to unite with the alumina to form the spinel molecule; but on the other hand the small amount of the alkalies and alkali-earth oxides do unite with a definite amount of the alumina to form the feldspar molecule. This again would seem to indicate, that in the presence of a certain amount of the alkali and alkali-earth oxides and silica that if there is enough silica present to unite with these oxides and the magnesia, there is no tendency for the magnesia to unite with the alumina to form any type of magnesia-alumina minerals.

Morozewicz* has recently described the formation of corundum from a number of magmas that were supersaturated with alumina. The supersaturated alumina-silicate magmas had the general composition $MeO \cdot mAl_2O_3 \cdot nSiO_2$ ($Me = K_2, Na_2, Ca$, and $n = 2-13$). In these magmas, on cooling, all the excess of alumina (over $m = 1$) separates out in the form of corundum crystals, this taking place when magnesia and iron were not present.

In these experiments, the silicates that were used to form the magmas were for the most part those of calcium, sodium and potassium. When alumina was present in them in excess of $m = 1$, it would separate out in the form of corundum; but when the magma was not saturated with alumina or m was equal to 1 or less, all the alumina was used up in the formation of double silicates. When magnesia was present, the excess of alumina united with the magnesia to form the mineral spinel, and if not enough magnesia was present to unite with all of

* Tschermak's Min. u. Petrogr. Mittheilungen, xviii, pp. 1–90 and 105–240, 1898.

the alumina, some corundum was formed. When there was an excess of silica, or n is greater than 6, the excess of alumina unites with the silica to form the mineral sillimanite and the over excess of alumina would separate out as corundum. When, however, magnesia and iron are present with the excess of silica and alumina, cordierite and spinel are the minerals formed.

From these experiments it would seem that where a magma is composed of silicates of the alkali and alkali-earth metals, sodium, potassium and calcium, that the alumina dissolved in this magma will to a certain point unite to form double silicates of alumina with these other bases; but when the ratio of alumina to these bases is greater than 1, the excess of alumina will separate out as corundum except when influenced as just described. Magnesia, it would seem, does not have as strong an affinity for the formation of silicates, and when present in the magmas, influences the separation of the alumina as a double salt of magnesia and alumina in the formation of the mineral spinel, or with an excess of both alumina and silica in the formation of the mineral cordierite.

In some earlier experiments, Morozewicz* has shown that alumina will readily dissolve in a molten magma having a composition approximately that of the basic magnesian rocks; and that upon cooling the alumina separates out as corundum and spinel.

There seems to be but little tendency for the alumina to unite with the magnesia to form the double silicates or with the magnesia alone to form the mineral spinel, $MgO.Al_2O_3$, except when there is an excess of MgO, when some spinel is formed, but the greater portion of the alumina separates as corundum. This would seem to show that the affinity of magnesia for silica in the formation of a silicate molecule is stronger than for alumina to form the aluminate molecule.

Summary.—From what has been observed in nature and from the experiments that have been made in the laboratory, it seems that the separation of alumina as corundum from molten magmas is dependent upon the composition of the chemical compounds that are the basis of the magma, upon the oxides that are dissolved with the alumina in the magma and upon the amount of alumina itself.

1. When the magma is a calcium-sodium-potassium silicate, no alumina held in solution by such a magma will separate out as corundum except when the ratio of the alumina to the other bases is more than 1 : 1 and the ratio of the silica is less than 6.

* Zeitschr. für Kryst., vol. xxiv, p. 281, 1895.

2. If magnesia and iron are present in the above magma, corundum will not form unless there is more than enough alumina to unite with the magnesia and iron.

3. When the magma is composed of a magnesium silicate without excess of magnesia, *all* the alumina held by such a magma will separate out as corundum.

4. Where there is an excess of magnesia in the magma just described, this will unite with a portion of the alumina to form spinel and the rest of the alumina will separate out as corundum.

5. Where there is chromic oxide present in a magma composed essentially of a magnesium silicate (as the peridotite rocks) and only a very little alumina and magnesia are present, these, uniting, separate out with chromic oxide to form the mineral chromite and no corundum or spinel are formed.

6. When peridotite magmas contain, besides the alumina, oxides of the alkalies and alkali-earths, as soda, potash and lime, a portion of the alumina is used in uniting with these oxides and silica to form feldspar.

7. There is a strong tendency for the alumina to unite with the alkali and alkali-earth oxides to form double silicates like feldspars, whether such silicates form the chief minerals of the resulting rock or are present only in relatively small amount. There is, however, but little tendency for the alumina to unite with magnesia to form double silicates when the magma is a magnesium silicate.

North Carolina Geological Survey, June, 1899.

SCIENTIFIC INTELLIGENCE.

I. Geology and Mineralogy.

1. *Canada; Summary Report of the Geological Survey Department for the year* 1898.—Among the items of general interest in the report are the following :

"The aggregate value of the production of minerals in Canada during the year 1897, as finally corrected and published, is $28,661,430, being an increase of about 27 per cent over that of the previous year. This is largely accounted for by the great development of gold mining, particularly in the Yukon district, the value of the gold produced being more than double that for 1896."

The Director examined the North Saskatchewan gold district, where much interest is taken in the use of dredging machinery to gather the river gravels. He reports regarding the occurrence of the gold in the "Saskatchewan gravels," that

"Gold in fine scales and particles, generally so minute as to require the employment of mercury in collecting it, is now known to occur on almost all the rivers running eastward from the Rocky Mountains, to the north of the International boundary, wherever these have been prospected. To the south of the Peace River, this gold, in any workable quantity, seems invariably to characterize a portion of the length of each of the rivers, giving out to the westward before the base of the Rocky Mountains is reached, and to the eastward along a less well defined line, but one probably due, in this case, to the local substitution of sand and clay banks and beds for the gravel bars of the upper and more rapid parts of the streams. The Peace River and the Liard, rising to the west of the Rocky Mountains proper, among rocks known to be auriferous, contain more or less gold throughout their lengths, or to points in their lower courses where the changed conditions of flow, above alluded to, render the collection of any minutely divided gold which they may still carry practically impossible. In other words, in these two rivers, and possibly also in some still farther to the north, a considerable part at least of the contained gold comes directly from their upper tributaries; while in the case of the Athabasca, the North Saskatchewan and other rivers farther to the south, the evidence at first sight appears to be entirely against the possible derivation of gold from the mountains to the westward."

"The result of recent observations appears, therefore, to indicate that the gold found in the Saskatchewan and other rivers of the Northwest has come in part from several sources, but has been derived chiefly from the crystalline rock of the Laurentian axis or plateau to the eastward or north-eastward, from which it has been transported with the fragments of these rocks that now form so conspicuous a part of the 'drift' of the Great Plains.

The recognition in late years of the Huronian as a distinctly gold-bearing formation, in itself goes far to establish the correctness of the hypothesis originally advanced on this subject, as rocks of this formation occupy considerable areas of the Laurentian plateau."

In these gravel deposits, six miles above Edmonton, bones of *Elephas primigenius* or *Americanus*, and of *Ovibos moschatus* have been discovered.

The overthrust faults in the Cretaceous coal-bearing district of Crow Nest Pass, on the eastern side of the Rocky Mountains, lead to the expectation of finding coal beds of Cretaceous age below Paleozoic limestone.

This peculiarity in the structure is adduced as an explanation of the petroleum occurring in rocks of which the surface is of probable Lower Cambrian age—between Crow Nest and South Kootenay passes in the southern part of the Rocky Mountains.

The borings for petroleum at Athabasca Landing have been continued, but without practical success. Mr. Fraser explains the hindrance to attempts to reach the sources of the petroleum in his report as follows:

"The bore at Pelican River had been stopped at 820 feet owing to the striking of an immense flow of gas, which made it impossible to work while it continued to flow with such force. It was thought that by the spring of 1898 it would have exhausted itself sufficiently to permit further boring, and to this end the casing, $4\frac{3}{8}$ inches in diameter, was left quite free and open to permit the escape of the gas. It was estimated that before a depth of 1000 feet was encountered the Devonian limestone would be pierced.

"Upon investigation in the early part of the present season, the flow of gas seemed to have very materially decreased; but upon operations being resumed, the seeming decrease was found to be in a great measure due to the closing up of the outlet at the bottom part of the casing by an asphalt-like mixture, composed of maltha, or petroleum tar, and sand. In fact, when boring operations were resumed on June 17th, the difficulty was found to be intensified by the accumulation of this asphalt-like maltha in the bottom of the bore.

"The rapid expansion of the gas produced a very low temperature, and this chilled and solidified the tar, or maltha, until it became as adhesive as wax. As the tools cut it loose the gas would carry it up through the bore, until from bottom to top it was almost one mass of sand and tar. The only way it could be extracted from the sand-pump was by heating the latter over a fire; even then very little could be got out at one time, it being so thick that it was almost impossible to force it up into the pump. I used different sorts of tools to cut it off the walls and clean it out, but the longer we worked at the bore the greater the quantity of tar accumulating on the sides of the casing and tools."

On the Pembina River in northern Alberta, Mr. McEvoy reports:

"Several outcrops of coal occur on the banks of the river, principally above the crossing. The coal has been on fire here years ago, and the overlying beds of clay and shale have fallen in, giving a very disturbed appearance to the locality. The white clay is partly burned to a pale red terra cotta. Half a mile above the crossing, on the east side, a seam of coal 17 feet 10 inches thick is exposed, of which the upper four feet is impure. On the opposite side there is a seam 13 feet thick, having four small partings of clay and carbonaceous shale, amounting in all to nine inches."

Dr. Adams in his study of the Grenville series in Monmouth reports important evidence of their sedimentary origin. He says:

"The study of the Grenville series in Monmouth showed beyond a doubt, that this series is a sedimentary one. It includes a great development of bedded white quartzites, evidently altered sandstones. The associated limestones also, that occur in heavy bands, and, as everywhere else in the Grenville series are in the form of white crystalline marbles, were in a few places along the line of the Irondale, Bancroft and Ottawa Railway, seen to hold little dark strings suggestive of remnants of the original limestone in a less altered condition. On this account, a careful search was made, which resulted in the discovery of two localities in which the limestone was almost unaltered, being very fine in grain and blue in color, and bearing a strong resemblance to the limestones of more recent formations. In such case the blue limestone is interstratified with the ordinary white coarse-grained marble of the Grenville series and passes into it, there being evidently portions of the limestones which have escaped metamorphism. These occurrences serve to dispose of any lingering doubts concerning the sedimentary origin of the limestone in question."

Silurian fossils were discovered about six miles from Canterbury station of the St. Andrews and Woodstock branch of the Canadian Pacific Railway, in a belt of rocks lying northwest of the granite area in western York. The rocks in question have hitherto been classified as Cambro-Silurian. They are partially altered gray slates. The fossils, according to Dr. Ami, indicate uppermost Silurian.

Along the shore of Grand Manitoulin Island, Lake Huron, a coralline limestone containing Lower Niagara fossils is reported by Dr. Ami.

Foot-prints of *Hylopus* and *Sauropus*, and other evidences of Carboniferous, have been reported from the fossiliferous strata along the north shore of the Basin of Minas.

"The bone-beds of McAra Brook, holding crustacean and fish remains, were again examined and additional examples were obtained of a *Pteraspis*, which appears to be new (the genus having been identified by Dr. A. S. Woodward), and fragments of cephalaspidian and acanthodian species, besides indications of

Pterygotus. The occurrence of *Pteraspis* and *Pterygotus* indicate that the beds at McAra Brook, above and below the post-road, are referable to the summit of the Silurian system or the lowermost portion of the Devonian."

Mr. Lambe reports the following Cretaceous fossils from Red Deer River district of Alberta, viz.:

"Considering first the fossils from the Belly River formation, the following provisional enumeration may be made of them:—

1. Chelonia:
   Fragments of the dorsal and ventral shield of *Plastomenus coalescens*, Cope.
2. Crocodilia:
   Parts of the rami of mandibles of a species of *Bottosaurus*, Agassiz.
3. Dinosauria:
   a. Numerous maxillæ and rami of mandibles and some of the principal bones of *Trachodon mirabilis*, Leidy.
   b. A maxilla with teeth, a separate tooth and a right ramus of a species of *Triceratops*, Marsh.
   c. Separate teeth and terminal phalanges of *Lœlaps incrassatus*, Cope.
   d. The upper part of the cranium of a species of *Nodosaurus*, Marsh." W.

2. *The Hereford Earthquake of December 17, 1896*; by CHARLES DAVISON, Sc.D., F.G.S., 303 pp. 8vo. Birmingham, 1899 (Cornish Brothers).—The careful investigation of the phenomena connected with an important earthquake is a laborious undertaking, but one which is of great value in its bearing upon our knowledge of earthquakes in general. In the case of the Hereford earthquake of Dec. 17, 1896, discussed in the present volume, Dr. Davison has shown unwearied energy in bringing the data together and much judgment and skill in drawing conclusions from them.

Of the several regions in Great Britain in which notable earthquakes have taken place, that of the occurrence here described, in the southeast of Herefordshire, is especially favorably situated for observation both geographically and as regards distribution of population. Here occurred also the earthquake of October 6, 1863, investigated by Mr. E. J. Lowe; while another, less violent, occurred in October 30, 1868. In 1896, however, the shocks were more numerous and severe, resulting in a very considerable amount of damage to buildings in the region of greatest intensity about Hereford. The effect was distinctly felt even at extreme points in the north and south of England and in Ireland.

The record of observations, contained in Part I (pp. 10–201) of the present volume, includes some 2902 separate accounts from 1943 places. The discussion of these data has made it possible to locate with accuracy the epicentral region with the isoseismal lines; also the isacoustic lines, or lines of equal sound intensity; the direction of the shocks at various points with the coseismal

lines. From the last the mean surface-velocity deduced between the first and third coseismals is 2955 feet per second.

In regard to the origin of the earthquake the conclusion is reached that it was due to a slip along a fault many miles in length having a general northwest and southeast direction and passing very near to Hereford. Further that there were two foci, one to the northwest near Hereford some eight miles in length, and a second, southeast and near Ross, about six miles in length. Between these there was an undisturbed portion of the fault about two miles in length. The fault-slip at the Hereford focus took place a few seconds before that at the Ross focus, hence the distinctly noted double series of vibrations. The position of the fault-line is located within the area of the Old Red Sandstone, but its exact geological relations have not been established, though it is plausibly connected with a well-recognized anticlinal in the neighborhood, bounded on three sides by faults, one of which has nearly the direction of the fault-line above mentioned.

3. *First Appendix to the Sixth Edition of Dana's System of Mineralogy*, by EDWARD S. DANA; pp. x and 75. New York, 1899 (John Wiley & Sons).—The sixth edition of Dana's System of Mineralogy was issued in the early part of 1892. In the seven years that have passed, the additions to mineralogical literature have been numerous and important; a summary of these is given in the Appendix now published, which thus completes the System to 1899. The prefatory note states as follows: "This Appendix contains, first of all, full descriptions of the species announced as new since the publication of the System. There are no fewer than one hundred and seventy names here included, and their place in the general scheme of classification adopted in the System is shown in the classified list given in the Introduction. In addition to the description of new minerals this Appendix is intended to contain also references to all important papers on mineral species published during the period named; with each reference is given a concise statement of its character, and so far as possible a summary of its contents. Since, however, the additions to mineralogical literature have been very numerous, it has been necessary, in order to keep this work within reasonable compass, to adhere rigidly to a system of extreme brevity of expression and conciseness of arrangement. All minerals named are, for convenience, placed in alphabetical order. . . . The Bibliography includes the full titles of prominent volumes published since 1891."

4. *Minerals of Mexico.*—Boletín del Instituto Geológico de Mexico, num. II, pp. 157, 4to. Mexico, 1898.—The latest number of the Bulletin published by the Geological Institute of Mexico contains a full and valuable catalogue of the mineral species found in the country, prepared by Prof. JOSÉ G. AGUILERA. In it the minerals are arranged according to the classification in Dana's System, the individual localities being given fully in connection with each species.

5. *Clays and Clay Industries of Pennsylvania.* (1) *Clays of Western Pennsylvania in Part:* by THOMAS C. HOPKINS.—This volume is issued as an appendix to the Annual Report of the Pennsylvania State College, for 1897. In it Professor Hopkins has given an interesting and instructive account of the various clays obtained in Western Pennsylvania, and further a description of the industries connected with their use. Numerous excellent illustrations accompany both parts of the subject.

II. MISCELLANEOUS SCIENTIFIC INTELLIGENCE.

1. *Eruption of Mauna Loa.*—Honolulu papers of July 8 to 10 contain accounts of the beginning of an eruption of the volcano of Mauna Loa, on Hawaii. Early in the morning of July 4th, one observer says, "an immense column of smoke and steam was seen rising from the crater of Mokuaweoweo. It was pierced through with the light from the fires beneath, until it glowed and shone like a column of fiery light, resplendent beyond description, and reflecting its burning glow over the whole heavens. The column seemed to be at least five miles in diameter, and rose to a tremendous height. On Tuesday, the column of fire had disappeared. In place of it was the equally impressive glow of the lava as it broke from the lower side of the crater several thousand feet lower down than the column of light had been, and was thrown upward to a wonderful height by the forces which were in action. On either side of the stream, whose surface of fiery red could be seen like a line of glowing molten metal, were two cones which had formed since the eruption began. It was from these that the lava was being ejected. It was thrown up in fiery cascades high in the air. These cascades, in falling, built up the cones, and the molten lava running off from these formed the stream flowing off toward Hilo. It would be hard to say how high these cones were, perhaps somewhere between 500 and 1000 feet high, and half a mile in diameter, and five miles apart." A later account mentions three lava streams, one in the direction of Hilo, another off through Kau to the southeast, and a third toward the crater of Kilauea.

Attention may be called in this connection to a paper by Mr. C. J. Lyons of Honolulu on "Sun Spots and Hawaiian Volcanoes" published in the April number of the *Monthly Weather Review.* The author gives a table of the years of minimum sun spots for the past century, with the dates of prominent volcanic eruptions of Kilauea or Mauna Loa, showing a striking correspondence between the times of the two phenomena. As pointed out by the editor of the *Review,* however, a more thorough investigation is needed to prove that the coincidence noted is due to a real causal connection.

The table referred to is as follows:

Years of minimum sun spots.	Years of most important lava flows or eruptions.
(?)	1790 (Kilauea Keoua eruption).
1799	1801 Hualalai.
1810	(?)
1823	1823 Mauna Loa.
1833	1832 Mauna Loa and Kilauea.
1843	1840 Kilauea. 1843 Mauna Loa.
1856	1852 1855 Mean 1856, Mauna Loa. 1859
1867	1868 Mauna Loa.
1878	1880-81 Mauna Loa.
1889	1887 Mauna Loa, south slope.
1900 (Probable)	----

2. *British Association.*—The coming meeting of the British Association for the Advancement of Science is to be held at Dover during the week beginning Sept. 13th. The occasion is expected to be a notable one and of international character, since the French Association meets at the same time at Boulogne and visits are planned for the two scientific bodies, on September 16 to Dover and on Sept. 21 to Boulogne.

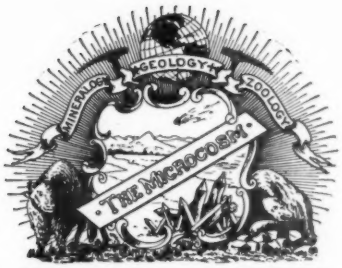
3. *American Association.*—The summer meeting of the American Association for the Advancement of Science was held at Columbus, Ohio, from August 21–26. An account of the meeting must be postponed to the following number.

4. *Sprachregeln für die Bildung und Betonung Zoologischer und Botanischer Namen;* von PAUL KRETSCHMER. 8vo, 32 pp. Berlin: 1899 (R. Friedländer & Sohn).—This pamphlet gives very full and carefully prepared grammatical rules for the formation and accentuation of zoological and botanical names and should be consulted by every naturalist interested in systematic nomenclature. S. I. S.

5. *Anleitung zur Darstellung Chemischer Präparate*, von Prof. Dr. H. ERDMANN. 2 Auflage; pp. 92. Frankfurt, 1899 (H. Bechold).—The first edition of Prof. Erdmann's little book on Inorganic Preparations appeared nine years ago, and has been very favorably received. This new edition, containing a few additional preparations, with numerous corrections and improvements, will, therefore, be welcomed by those who are interested in teaching experimental chemistry. H. L. W.

OBITUARY.

Sir WILLIAM HENRY FLOWER, K.C.B., F.R.S., the distinguished Zoologist, late Director of the Natural History Department of the British Museum, died on July 1 in his sixty-eighth year. His contributions to zoology, anatomy and anthropology were numerous and important, and in his capacity of museum curator he performed a work of great and lasting value, the influence of which has been widely felt in other institutions at home and abroad.



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Llano del Inca (3),	8 to 12 "	Mt. Joy (7)	8 to 12 "
Fayette Co (4),	3 to 8 "		

(1) Described in this Journal Sept., 1895. (2) Ibid Nov., 1887. (3) Proc. Roch. Acad. Sci. Vol. 1. (4) This Journal Aug., 1888. (5) Ibid July, 1893. (6) Ibid June, 1894. (7) Ibid Nov., 1892.

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MURPHY.

This aerosiderite was found about April, 1899, near Murphy, Cherokee County, N. C., and was obtained in its entirety by us. We have cut it parallel to the larger end; so that each slice gives a complete section through the meteorite. These are etched upon one side and show well marked Neumann figures; small troilites also appear in all. We offer the following sections:

202 grammes	$30	576 grammes	$ 80
370 grammes	55	1054 grammes	140
502 grammes	70	1540 grammes (end piece)	200

The smaller end of this iron had been broken off with a hammer by the original finder. This piece has been cut vertically, giving two broad sections showing the exterior of the iron on one surface. These pieces we offer as follows:

259 grammes	$40	293 grammes	$45

One section shows upon the unetched surface the fractured face, upon which the crystalline structure is well shown. **217 grammes, $40.**

One of the smaller pieces has been reserved for re-sectioning, and will be cut to order at the following rates:

10 grammes or less, per gr...	$0.50	50 grammes	$12
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This meteorite will be noted in a forthcoming issue of the American Journal of Science.

NESS COUNTY.

Since announcing this find in the American Journal of Science, Vol. VII, p. 233, several other stones of the same fall have been found and have come into our hands. At present we have, besides a fraction of the original stone, but a single specimen.

This is sub-pyrimidical in form, without a single break in the crust covering it, and weighs 1570 grammes. It is a handsome specimen and will be sold for $125. Of the original stone we can still furnish the following pieces with both cut and broken surfaces:

4 grammes	$2.75	20 grammes	$13.00
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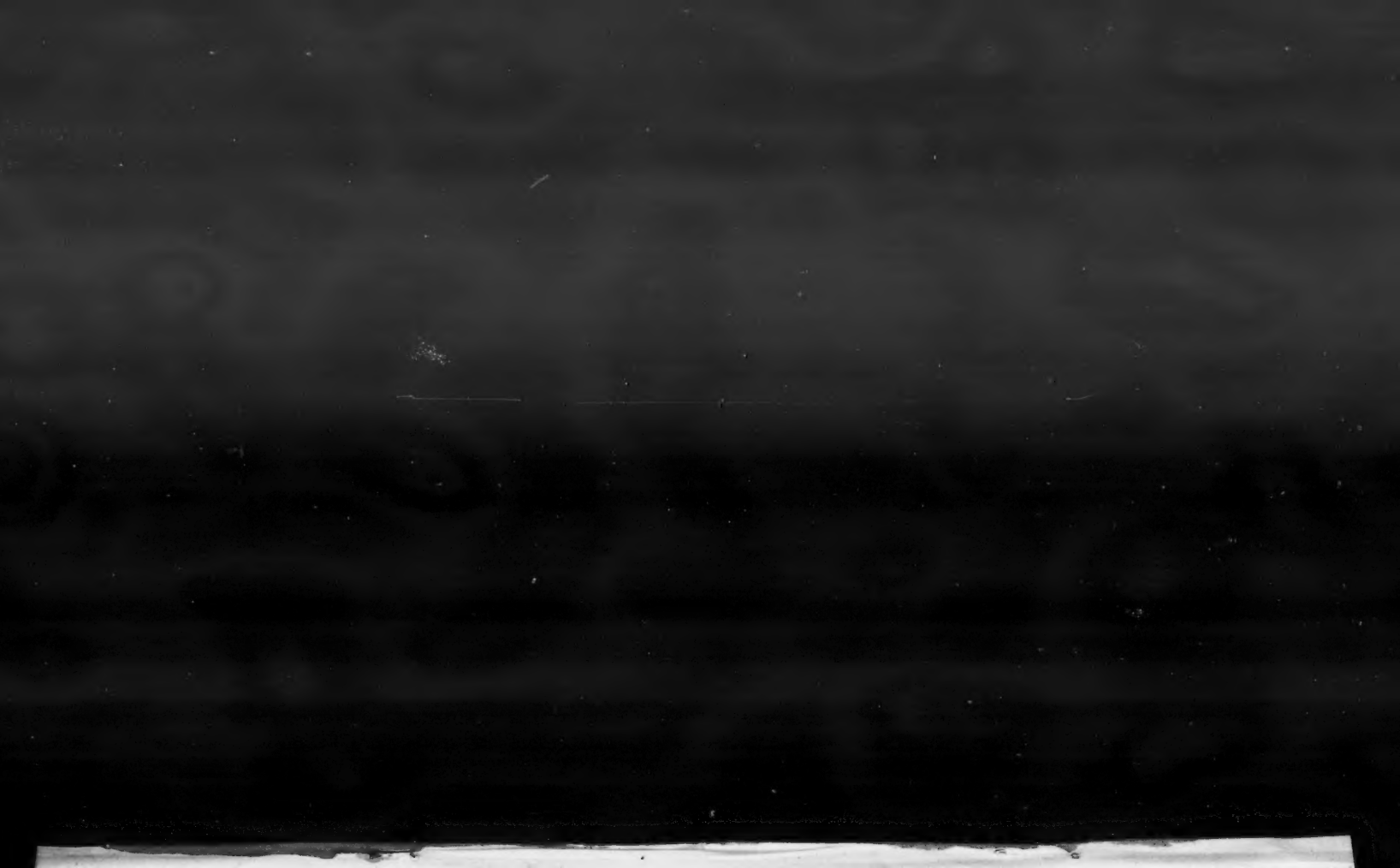
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